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Microstructure and Phase Evolution of Ni_2FeGa Heusler alloy extended to Different Degrees of Undercooling.

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Abstract: The Ni_2FeGa Heusler alloy is synthesized by arc melting in argon atmosphere. It shows two phase microstructure, γ-phase (disordered fcc) and Austenite (ordered bcc, L_{21}). Phase identification and microstructural characterization were carried out using XRD, SEM and TEM. Solidification at various undercoolings upto 215 °C was performed using flux undercooling technique. B_2O_3 was used as the flux that provides an inert atmosphere and isolates the molten pool from the quartz tube. The solidified microstructure of the undercooled samples were analyzed and the result indicates γ-phase to be the primary phase to form. The samples are also textured. XRD patterns indicate different texture at different undercoolings. Possible mechanisms for such changes will be discussed. The competitive nucleation mechanism can not also be ruled out as the SEM micrographs show the globular morphology of γ-phase likely due to defragmentation of primary dendrites. Thermal analysis by DSC shows incongruent melting of Ni_2FeGa Heusler alloy which strengthen the argument of poor nucleation ability of L_{21} ordered intermetallic austenite phase as compared to primary γ-phase. Up to achieved undercooling limits, γ-phase forms as the primary phase competitively with the L_{21} ordered phase. Studies indicate that competitive nucleation mechanism is a likely mechanism to explain the phase selection.

Introduction

Magnetic Shape Memory (MSM) alloys have gained lot of interest because of their fast response to magnetic field. Applications like actuation and sensor for efficient use requires quick response to external applied force. That gives an advantage to MSM alloys over conventional temperature driven shape memory alloys. Ferromagnetic Heusler alloys shows unique magnetoelastic coupling which gives rise to giant magneto-elastic strain [1] and magneto-caloric effect [2]. Among Heusler alloys, NiMnGa has been studied most widely because of it's large MFIS 9.5% [1, 3]. But the main issue is the fragile nature of this alloy. So, many researchers have tried to induce ductility and toughness by adding fourth element like (Fe, Co, etc) into the composition [4, 5] or replacing Mn with Fe or Co [6, 7]. Other Ni-Mn-Ga like systems Ni-Mn-Z (Z = In, Sn, Al) [8-11] also have been reported showing shape memory effects. The enhancement of ductility and toughness of the heusler alloy has been a positive improvement of Fe-addition.

Solidification studies on Heusler system are limited. Particularly, the phase evolution in Ni-Fe-Ga Heusler system is not well understood. Solidification microstructure of the alloy as a function of undercooling is necessary to understand the phase evolution and microstructure development in material [12]. Non-equilibrium solidification process using flux undercooling technique gives the possibility to undercool the sample well below it's liquidus temperature (1290 °C) by eliminating the heterogeneous nucleation sites [13]. This undercooling provides sufficient driving force required for the formation of metastable phases which may not be present in equilibrium phase diagram [14-15]. In Ni-Fe-Ga alloy system, disordered γ-phase (fcc) forms competitively along with ordered (L_{21}) austenite phase for the composition at/near Heusler stoichiometry [16]. In this article, the microstructure and phase evolution of Ni_2FeGa Heusler alloy was studied as a function of the degree of undercooling.

Experimental Details

Ni_2FeGa Heusler alloys were prepared by choosing the nominal stoichiometry X_2YZ. High purity (99.99%) Ni, Fe and Ga elements were mixed together as per their atomic % (by converting into weight %). The alloys were cast into button shape (approx. 1g) using chilled Cu-hearth in argon atmosphere by arc melting in vacuum (10^{-5} mbar) and backfilled with argon. The alloys were melted four times to promote compositional homogeneity, everytime rotating it upside down. The solidification path of Ni_2FeGa Heusler alloy was investigated by adopting flux undercooling technique. The sample was placed inside a quartz tube (dia 10/12 mm, length 65mm, curved bottom) along with the flux. The experiment was performed using induction technique in ambient condition. The flux, boron trioxide (B_2O_3) was used as the reagent to provide an inert atmosphere by isolating the molten pool from the quartz tube. A two color infrared colored pyrometer was employed to record the time – temperature profile during both heating and cooling cycles to understand the solidification behavior. Number of heating and cooling cycles were performed to obtain the desired undercooling. Phase identification and microstructural characterization were carried out using XRD, SEM and TEM. X-ray diffraction was used as the primary technique for structural characterization by X'Pert PANalytical®. The microstructure and their composition was analyzed by SEM Quanta 200® with EDX attachment. The structure of the phases present in the alloy was confirmed by TEM (CM 12®) along with the XRD results.

Results and Discussion

The structure of the Ni_2FeGa Heusler alloy was identified and indexed as shown in the XRD pattern in Fig.1. It shows a two phase structure, (γ-phase, disordered fcc) and Austenite (ordered bcc, $L2_1$ structure). The lattice parameters of fcc and $L2_1$ structures are 0.360 nm and 0.570 nm respectively. The similar structures have been reported elsewhere [17]. The superlattice reflections (111) and (200) are absent in XRD pattern. For the Heusler stoichiometry X_2YZ, if any anti-site disorder is present between Y and Z lattice positions, (111) peak can be absent. Similarly, (200) peak intensity can be much reduced as compared to fundamental reflection (220) due to the anti-site disorder between X and Y lattice positions [18]. The superlattice peaks are very weak and their disappearance could be due to the almost similar atomic scattering factors [19]. The SEM image of Ni_2FeGa Heusler alloy is shown in Fig.2. The γ-phase forms as the primary phase that the dendritic morphology. Compositional analysis of the phases by EDX attached to scanning electron microscope shows γ-phase is rich in Fe-content ($Ni_{50}Fe_{32}Ga_{18}$) and $L2_1$ phase is very close to stoichiometry ($Ni_{50}Fe_{23}Ga_{27}$), but Ga-content is higher than Fe. It is believed that in γ-phase, few Fe-lattice positions are occupied by Ga. This substitution is random and forms disordred fcc structure as characterized by XRD shown in Fig.1. But in case of austenite phase, the atoms preferentially occupy their respective lattice positions and form an ordered crystal structure.

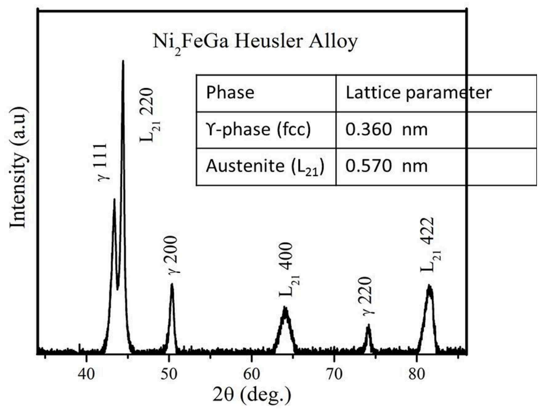


Fig.1: XRD pattern of Ni_2FeGa Heusler alloy.

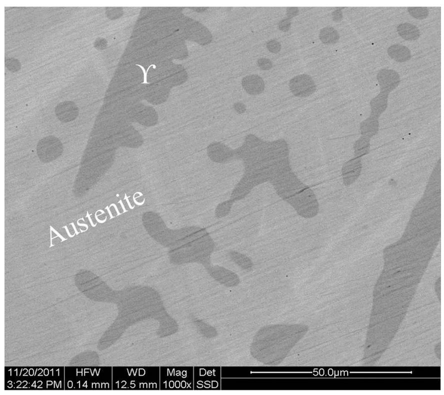
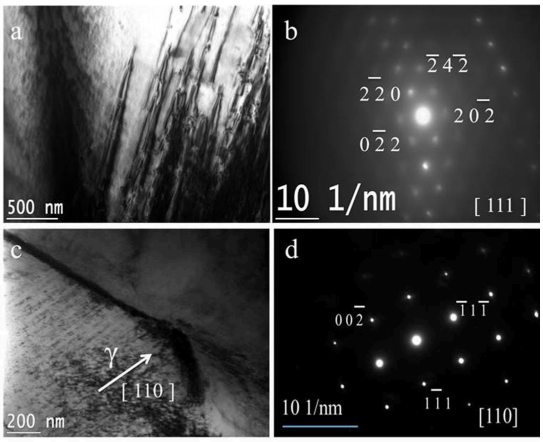


Fig.2: SEM image of Ni_2FeGa Heusler alloy.

The dendritic morphology of primary γ-phase is prominently visible in bright field image (bf-image, Fig.3a) and the corresponding SAED pattern was indexed as disordered fcc structure with [111] zone axis Fig.3b. The second bf-image in Fig.3c shows a thick and clearly distinct grain boundary between γ-phase and austenite. SAED pattern was taken from both the sides of grain boundary and shown in Fig.3(d, f). The γ-phase structure was indexed with zone axis [110] and austenite with [110]. The appearance of weak spots are assigned to the superlattice reflections from (111) and (200) crystal planes [20].



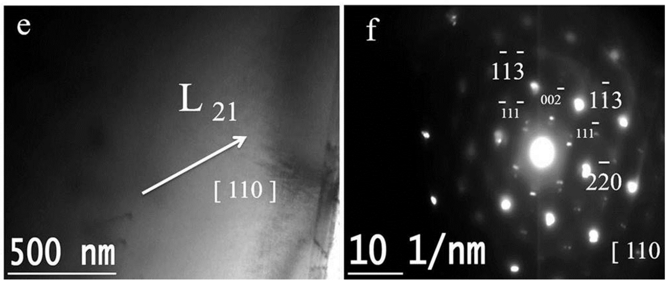


Fig.3: TEM images of Ni_2FeGa Heusler alloy (a) dendritic γ-phase, (c) bf-image of both phases along with phase boundary, (b,d) corresponding diffraction of γ-phase, (e) TEM bf-image of L_{21} phase and (f) corresponding diffraction

The presence of two phases in the microstructure for a large extent of undercooling show that alloys near the stoichiometric composition of Ni_2FeGa lie in two phase region (γ + Austenite). It was not possible to obtain a phase-pure microstructure in this alloy upto an undercooling of 215 °C. Different undercoolings upto maximum 215 °C were obtained and presented in Fig.4. For convenience, the alloys are named GF-1 to GF-7 with increasing undercooling. This nomenclature is used for the rest of the manuscript for easy reference. The XRD results are shown in Fig.5, again shows both the phases (γ + Austenite) are present upto achieved undercooling. The formation of texture during solidification is very likely in Ni_2FeGa Heusler alloy that is revealed from XRD pattern. The high angle peaks of γ-phase (200) in GF-2,3 and 6 alloys and (220) peak in GF-7 alloys are quite intense. That gives information about the solidification texture in the material due to limited number of grains in the microstructure [21].

It is likely that owing to the higher Fe-content, the dentritic phase is similar to the γ-phase in Ni-Fe alloy system [22]. Atomic size of both Ga (0.122 nm) and Fe (0.124nm) are comparable, their difference in atomic size is less than 2%. It could be possible that Ga makes substitutional solid solution in Ni-Fe crystal lattice, preferably occupies the Fe-lattice positions as the alloy composition is $Ni_{50}Fe_{25}Ga_{25}$ and the other phase forms is close to the stoichiometry. From this result, it is concluded that partitioning of Fe and Ga takes place while phases are forming. Fe seems to stabilize γ-phase while Ga does the same for austenite. Thus, the Heusler composition $Ni_{50}Fe_{25}Ga_{25}$ lies in the two phase region that forms during solidification.

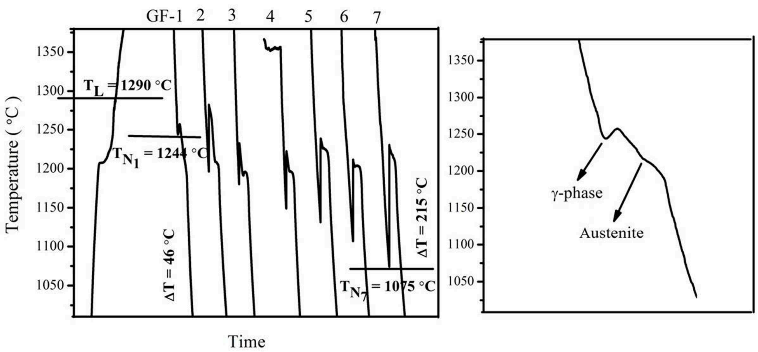


Fig.4: Different undercooling curves of Ni_2FeGa Heusler alloy, inset shows the two stage solidification

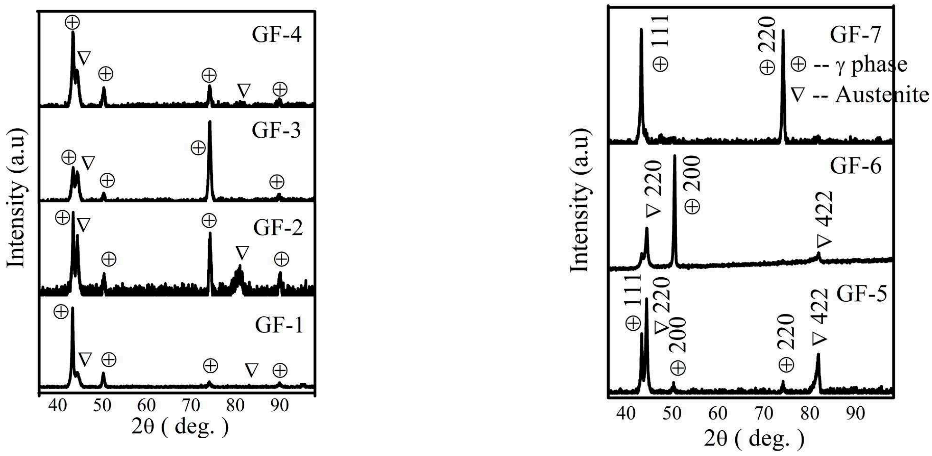


Fig.5: XRD Patterns of all undercooled Ni_2FeGa Heusler alloys

For the undercoolings achieved upto 215 °C, though both the phases (γ + Austenite) are present, the fraction of γ-phase keeps increasing with undercooling [7,26]. The same has been correlated with the properties measurement like Vickers hardness and magnetization. The Hardness value decreases with increasing undercooling as γ-phase fraction increases (Fig.6, inset(a)). The improvement of ductility is the striking difference in case of Ni-Fe-Ga Heusler alloy than from the Ni-Mn-Ga. The same has been attributed to the γ-phase as the disordered fcc structure provides less hindrance for the dislocation movement than corresponding ordered structure [27]. Again, the increasing γ-phase fraction with undercooling is quite evident from M - H measurement at room temperature which shows the magnetization increases as sample undercooling increases (Fig.6).

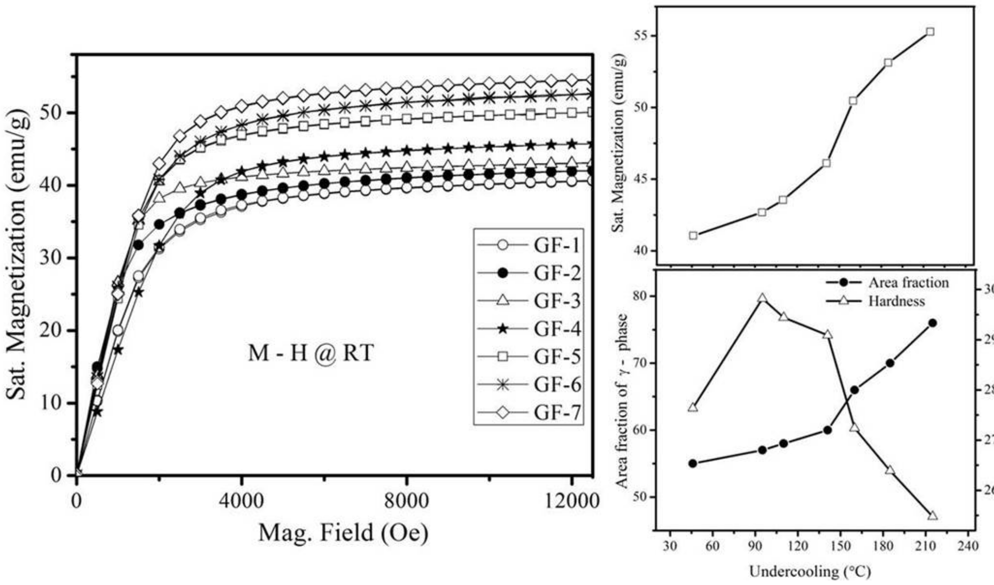


Fig.6: Magnetization curves of all undercooled Ni_2FeGa Heusler alloys

The increase in magnetization value is correlated to the increasing γ-phase fraction as the saturation magnetization of γ-phase is higher than that of Austenite. In Heusler alloy X_2YZ, generally, the magnetic moment is localized in Y-site and exchange interaction takes place between Y-atoms mediated by Z-atoms [28]. Similarly in Ni_2FeGa, the magnetic properties are attributed to the Fe-atom in Y-site and Fe-Fe inter-atomic distance also plays an important role. In Austenite, the exchange interaction is modified by the presence of anti-site disorder as Fe-atoms are partially replaced by Ga-atom, which decreases the magnetic moment value. But in γ-phase, the localized magnetic nature is lost as Fe is not restricted to any particular lattice site. The number of Fe-atoms is higher than Ga-atoms which increases the exchange interaction and consequently the magnetic moment. Thus, the magnetic measurement agrees well with our observation of increasing γ-phase fraction with undercooling.

The increasing γ-phase fraction is likely due to higher nucleation rate as undercooling increases and that leads to the defragmentation of dendrites into more rounded morphology of γ-phase. Consequently, the second phase appears from the inter-dendritic liquid as the solidification progresses. The γ-phase forms as primary phase predominantly by taking more Fe-content and rejecting Ga into the melt. Thus, the melt becomes compositionally instable w.r.t γ-phase having less Fe and more Ga content and solidifies into a different phase as Austenite with composition close to Heusler stoichiometry. Nucleation of Austenite is delayed as long γ-phase starts forming that shows its poor nucleation ability as compared to the primary phase. The two stage solidification behavior also has been depicted in the inset shown in undercooling curve. Therefore, it is believed that phase selection happens by competitive nucleation mechanism which prefers γ-phase as the primary phase and Austenite as secondary.

Summary

The phase and microstructural evolution of Ni_2FeGa Heusler alloy was studied using flux-undercooling technique. The microstructure consists of two phase (γ + Austenite) for the whole range of undercooling achieved upto 215 °C, which is similar to the as cast alloy. But, γ-phase forms increasingly as undercooling increase and the same has also been confirmed by hardness and magnetic measurement. It is assumed that γ-phase nucleates competitively with L_{21} phase. Phase separation takes place due to Fe and Ga concentration difference in the respective phases. Once γ-phase has formed, the nucleation of ordered Austenite phase is delayed and starts nucleating from the melt continuously throughout as the inter-dendritic phase when the condition becomes favorable.

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