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# Hot deformation behavior of Ni—Fe—Ga-based ferromagnetic shape memory alloy — A study using processing map



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#### ABSTRACT

Ni–Fe–Ga-based alloys form a new class of ferromagnetic shape memory alloys (FSMAs) that show considerable formability because of the presence of a disordered fcc  $\gamma$ -phase. The current study explores the deformation processing of this alloy using an off-stoichiometric Ni<sub>55</sub>Fe<sub>19</sub>Ga<sub>26</sub> alloy that contains the ductile  $\gamma$ -phase. The hot deformation behavior of this alloy has been characterized on the basis of its flow stress variation obtained by isothermal constant true strain rate compression tests in the 1123–1323 K temperature range and strain rate range of  $10^{-3}$ –10 s<sup>-1</sup> and using a combination of constitutive modeling and processing map. The dynamic recrystallization (DRX) regime for thermomechanical processing has been identified for this Heusler alloy on the basis of the processing maps and the deformed microstructures. This alloy also shows evidence of dynamic strain-aging (DSA) effect which has not been reported so far for any Heusler FSMAs. Similar effect is also noticed in a Ni–Mn–Ga-based Heusler alloy which is devoid of any  $\gamma$ -phase.

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## 1. Introduction

Heusler Ni-Mn-Ga-based ferromagnetic shape memory alloys (FSMAs) are known to exhibit large magnetic-field-induced-strain (MFIS) reaching up to 10% [1] and have emerged as one of the important materials for possible applications in sensors and actuators, in particular for high frequency actuators. However, lack of ductility of Ni-Mn-Ga-based alloys often limits its technological potential, especially in polycrystalline form. Poor grain boundary cohesion is considered to be the main reason behind this. Similar problem is also encountered in structurally analogous Ni-Al-based system, where considerable formability could be achieved by incorporating a disordered fcc  $\gamma$ -phase in the B2-matrix [2,3]. Oikawa et al. [4] first identified the possibility of utilizing an identical method in case of the ferromagnetic Heusler alloys and introduced this new class of Ni-Fe-Ga-based two-phase ductile FSMAs. This alloy system has since attracted attention of a large number of investigations [5–9]. Similar to Ni–Al system, the brittleness of the Ni-Fe-Ga alloys can largely be overcome by introducing a disordered fcc  $\gamma$ -phase. Moreover, these alloys also are

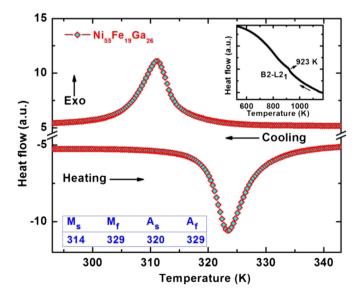
found to be amenable to thermo-mechanical processing (TMP) [10], which can potentially be exploited for achieving a favorable texture that imparts desired properties. There have also been studies on a few other similar two-phase FSMAs such as Co-Ni-Al [11] and Ni-Mn-Ga-Fe [12]. Ternary Ni-Fe-Ga alloys with compositions close to the Ni<sub>2</sub>FeGa stoichiometry show thermoelastic martensitic transformation (MT) with characteristics quite similar to Ni<sub>2</sub>MnGa alloys; they also show a strong coupling between the structural and the magnetic transitions [13,14]. The parent austenite phase is ordered cubic L2<sub>1</sub> that changes to B2 above the L2<sub>1</sub>-B2 order transition temperature [10]. Three different types of martensite crystal structures, both modulated (10M and 14M) [4,8,9,13,14] and nonmodulated (NM) [15], are known to occur in this system. Apart from that, a 6-layer martensite has also been reported [5,6,16]. There have been a few studies on the stress-induced martensitic transformation and inter-martensitic transformation as well [7,17,18]. In fact, the phase and microstructure in this system are decided to a large extent by the chemical composition since it has strong influence on both the Curie temperature  $T_C$  and MT temperatures [4,14]. In addition, aging treatment also plays key role in changing both MT temperatures and the microstructure of Ni-Fe-Ga alloys [9,10,19]. In particular, the volume fraction of the ductile  $\gamma$ -phase and hence its mechanical behavior, can be tailored by tuning both the composition and aging treatments [7,14].

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Polycrystalline FSMAs would become attractive – from the technological application point of view - if they are favorably textured [20,21], which can be achieved through high temperature TMP. This has been attempted by a number of researchers on the more popular Ni-Mn-Ga-based alloys over the years and with varying degree of successes [22-27]. These include hot-rolling [22,23], hot-forging [24,25], hot-extrusion [26] and highpressure-torsion [27]. As for the Ni-Fe-Ga system, we have earlier demonstrated substantial hot-rollability in a two-phase alloy of the following composition: Ni<sub>55</sub>Fe<sub>19</sub>Ga<sub>26</sub> [10]. In another study, Maziarz et al. [28] have reported hot-rolling of a similar twophase FSMA in Co-Ni-Al system. However, and in general, detailed studies on the high temperature deformation behavior of FSMA are lacking. In particular, even though the two-phase Ni-Fe-Ga-based alloys are known for their improved ductility, there has not been any systematic study on their formability and the resultant evolution of deformation microstructure. Keeping this in view, this work sets out to elucidate the hot deformation behavior of Heusler Ni-Fe-Ga-based FSMA using a two-phase ternary alloy Ni<sub>55</sub>Fe<sub>19</sub>Ga<sub>26</sub>, which exhibits remarkable hot-workability [10]. For this purpose, we examine the high temperature deformation response over a wide temperature, T, range, where ordered B2 is stable, and with strain rates,  $\dot{\varepsilon}$ , varying from  $10^{-3}$  to  $10 \text{ s}^{-1}$ . The experimental data were analyzed by recourse of the processing maps approach pioneered by Prasad and co-workers so as to identify the optimum combination of T and  $\dot{\varepsilon}$  for hot working of the alloy. This was combined with the study on the evolution of the two-phase microstructure of Ni<sub>55</sub>Fe<sub>19</sub>Ga<sub>26</sub> upon hot deformation and the role of disordered  $\gamma$ -phase in it.

### 2. Experimental procedures

Pancake-shaped ingots of the ternary alloy: Ni<sub>55</sub>Fe<sub>19</sub>Ga<sub>26</sub>, with a typical weight of 350 g each, were made by vacuum arc melting high purity (99.99%) elements in appropriate proportions. Homogeneity was ascertained by remelting the ingots multiple times. They were then solutionized under an inert atmosphere at 1353 K for 24 h followed by low temperature annealing at 873 K for 5 h to promote atomic ordering. The ordering temperature was determined with the aid of high temperature differential scanning



**Fig. 1.** DSC plot of  $Ni_{55}Fe_{19}Ga_{26}$  alloy showing the martensitic transformation, relevant temperatures are tabulated in the lower inset; upper inset shows the B2-L2<sub>1</sub> order-disorder transition.

calorimetry (DSC), which indicated to a B2-L2<sub>1</sub> transition at 923 K [inset of Fig. 1]. After annealing, the ingots were air cooled so as to enable  $\gamma$ -phase precipitation. Detailed characterization of these alloys has been carried out using optical, scanning and transmission electron microscopies for microstructural analysis, electron probe micro-analysis (EPMA) for chemical composition analysis, X-ray diffraction (XRD) for phase identification, and DSC. Samples for metallography were etched using an aqueous solution of FeCl<sub>3</sub> in HCl. High and low-temperature isochronous DSC experiments were performed using Setaram and Mettler-Toledo instruments, respectively, at a rate of 10 K/min in Ar atmosphere. Prior to the compression tests, phase-specific microhardness tests were carried out using a Struers Duramin microhardness tester for the following three phases: martensite, L2<sub>1</sub> and  $\gamma$ -phase precipitates.

Compression test specimens with dimensions: 6 mm diameter and 9 mm height were extracted from the solutionized ingot using electric discharge machining. Spark drilling was used to make a tiny hole in each one of them for insertion of thermocouple. Compression tests were conducted under isothermal conditions at constant  $\dot{\varepsilon}$  of  $10^{-3}$ ,  $10^{-2}$ ,  $10^{-1}$ , 1 and 10 s<sup>-1</sup> and at T of 1123, 1173, 1223, 1273 and 1323 K in a servo-hydraulic machine (DARTEC, UK) equipped with split-type resistance heating furnace. In order to reduce friction between specimen and anvil, surface of the specimen were lubricated with graphite powder. The samples were deformed up to a reduction of 50% height, which corresponds to a true strain,  $\varepsilon$ , of ~0.7. The temperature was monitored using a thermocouple attached to the specimen with an accuracy of temperature control within +2 K. Specimens were soaked for 5 min prior to the test. After deformation, samples were sectioned parallel to the compression axis for microstructural characterization. The loadstroke data obtained from the compression test are converted into true stress-true strain curves. The adiabatic temperature rise in specimen during deformation was found to be negligible, hence, not taken into account.

#### 3. Results

#### 3.1. Initial microstructure and phase analysis

Martensitic transformation in  $Ni_{55}Fe_{19}Ga_{26}$  alloy is shown in the DSC plot in Fig. 1; the inset shows the L2<sub>1</sub>-B2 order-disorder transformation at 923 K. As expected from the DSC results, the as-solutionized  $Ni_{55}Fe_{19}Ga_{26}$  alloy shows a two-phase microstructure, with the cubic  $\gamma$ -phase precipitates embedded in a martensite matrix, as shown in Fig. 2(a) and (b). The matrix is coarse-grained, with a typical grain diameter of ~500  $\mu$ m (Fig. 2a). The precipitates are observed both at the grain boundaries as well as at the grain interiors, and are ~19 vol.%, which is determined on the basis of EBSD analysis [10]. Chemical compositions of these two phases are determined by EPMA, details of which are given in an earlier report [29]. Martensitic matrix in this ternary alloy is found to be richer in Ga but leaner in Fe, as compared to the second-phase precipitates.

XRD and TEM analyses confirm the majority martensite phase to be an internally-twinned tetragonal non-modulated (NM) type as displayed in the inset of Fig. 2(b). A few extra peaks observed in XRD analysis are identified as reflections from a modulated 14M martensite phase [29]. Incidentally, the minor fraction of the modulated 14M martensite in this alloy could not be detected in TEM because of the length scale involved. Detailed phase analysis of this alloy is reported elsewhere [10,29].

Results of the phase-specific microhardness tests (Fig. 3a and b) are listed in Table 1. It may be noted here that the Ni $_{55}$ Fe $_{19}$ Ga $_{26}$  alloy is aged at 873 K to obtain L2 $_1$  matrix in place of the martensite [30]. Interestingly, the martensitic matrix and  $\gamma$ -phase precipitates have similar hardness at room temperature. L2 $_1$  matrix, on the other

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