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Development of Magnetic Actuator Materials for Energy Harvesting

Final Report

R.A. Dunlap, T.D. Hatchard, J.S. Thorne, J.M. Gaudet, S.E. Flynn, and M. Maillet

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Contract Project Manager: Dr. R.A. Dunlap, 902-494-2394
PWGSC Contract Number: W7707-063371/001/HAL
CSA: Dr. Shannon P. Farrell, Defence Scientist, 902-427-3437

Defence R&D Canada – Atlantic

Contract Report
DRDC Atlantic CR 2009-117
September 2012

Canada

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Defence R&D Canada – Atlantic

Contract Report
DRDC Atlantic CR 2009-117
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Abstract

Magnetostrictive alloys are novel 'smart' materials that exhibit dynamic coupling of magnetic and mechanical properties. The dynamic magnetic-mechanical coupling phenomenon may make magnetostrictive alloys useful for transforming kinetic energy into magnetic energy to be harvested for electrical energy (and vice versa). This offers the potential for magnetostrictive alloys to be useful for development of efficient, cost-effective transducer, actuator, smart structure, vibration dampening and energy harvesting materials for military applications.

Several low-cost processing approaches that produce highly-textured thin-form magnetostrictive Fe-Ga alloys were found to be promising for energy harvesting applications. Dalhousie University was contracted to evaluate several techniques for the production of magnetically active alloys and to evaluate the resultant thermal, magnetic and crystallographic properties of the alloys. This document summarizes the principal results from the final two years of the project and focuses primarily on development of Fe-Ga magnetostrictive alloys. Mechanical alloying (via ball milling) and rapid quenching (ribbon casting) were shown to be cost-effective methods for producing Fe-Ga compositions. The magnetic and crystallographic structures were fully characterized and show unique properties. ^{57}Fe Mössbauer hyperfine fields were shown to be particularly sensitive to atomic scale clustering (as a precursor to ordering). Highly-oriented feedstock Fe-Ga rods have been produced using the cost effective suction extraction method. Fe-Ga rods provided excellent feedstock material that improved the quality, i.e., <100> texture, of DRDC's fabricated Fe-Ga magnetostrictive wire.

Résumé

Les alliages magnétostrictifs constituent de nouveaux matériaux « intelligents » qui présentent un couplage dynamique des propriétés magnétiques et mécaniques. Le phénomène de couplage magnétique mécanique pourrait faire des alliages magnétostrictifs des matériaux utiles pour transformer l'énergie cinétique en énergie magnétique pouvant être récupérée pour produire de l'énergie électrique (et pour exécuter le processus inverse). Les alliages magnétostrictifs pourraient donc se révéler très utiles dans la mise au point des matériaux efficaces et peu coûteux qui servent à fabriquer des transducteurs, des actionneurs, des structures intelligentes et des dispositifs d'amortissement des vibrations et de récupération d'énergie ayant des applications militaires.

Les résultats des études indiquent qu'il existe plusieurs méthodes de production peu coûteuses d'alliages magnétostrictifs de fer et de gallium (Fe Ga) sous forme de corps minces et fortement texturés qui semblent prometteurs en matière de récupération d'énergie. Un contrat a été conclu avec l'Université Dalhousie afin qu'on y effectue l'évaluation de plusieurs techniques de production d'alliages magnétiques actifs et la détermination de leurs propriétés thermiques, magnétiques et cristallographiques. Le présent document contient un résumé des principaux résultats obtenus au cours des deux dernières années du projet, plus particulièrement ceux ayant trait aux travaux de mise au point d’alliages de Fe Ga magnétostrictifs. Les résultats démontrent que l’alliage mécanique (par broyage à boulets) et la trempe instantanée (coulage de bandes) constituent des méthodes rentables de production d’alliages de Fe Ga de diverses compositions. La caractérisation poussée des structures magnétiques et cristallographiques des produits indique qu’ils possèdent des propriétés exceptionnelles. L’analyse des champs hyperfins par spectrométrie Mössbauer du 57Fe montre qu’ils sont particulièrement sensibles à la présence de clusters à l’échelle atomique (comme éléments précurseurs à l’obtention d’une structure ordonnée). Il a été possible de produire des tiges brutes en Fe Ga à forte symétrie d’orientation en employant la méthode d’extraction par aspiration, qui est très rentable. Les tiges en Fe Ga constituent d’excellentes matières premières qui permettent d’accroître la qualité, c.-à-d. la texture cristallographique <100>, des fils de Fe Ga magnétostrictifs fabriqués dans les installations de RDDC.

Executive summary

Development of Magnetic Actuator Materials for Energy Harvesting: Final Report

R.A. Dunlap, T.D. Hatchard, J.S. Thorne, J.M. Gaudet, S.E. Flynn and M. Maillet; DRDC Atlantic CR 2009-117; Defence R&D Canada – Atlantic; September 2012.

Introduction: Magnetostrictive alloys exhibit a dynamic coupling of magnetic and mechanical properties. This coupling enables the transformation of kinetic mechanical energy into magnetic energy and, when properly configured, electrical energy (and vice versa). Therefore, magnetostrictive alloys have potential for use as cost-effective transducer, actuator, smart structure, vibration dampening and energy harvesting materials for military applications. The Dockyard Laboratory (Atlantic) undertook a 3 year Technology Investment Fund project to research and develop magnetostrictive alloys for energy harvesting applications.

Dr. Dunlap of Dalhousie University (Department of Physics and Atmospheric Science) was contracted to evaluate fabrication and processing approaches for production of magnetically active alloys and to evaluate the resultant thermal, magnetic and crystallographic properties. A previous contract report (DRDC Atlantic CR 2007-336; January 2008) summarized the principal results from the initial year of this project and had helped defined the research plan for the remaining 2 years.

Results: This report documents the significant outcomes from the final two years of the project and focuses primarily on development of Fe-Ga magnetostrictive alloys. Mechanical alloying (via ball milling) and rapid quenching (ribbon casting) were shown to be cost-effective methods for producing Fe-Ga compositions. The magnetic and crystallographic structures were fully characterized and show unique properties. A cost-effective approach for fabrication of Fe-Ga feedstock material was developed and enabled production of the <100> textured Fe-Ga magnetostrictive wire.

Significance: After completion of this research, DRDC is better positioned to research, assess and advise on the significance and technical readiness of novel ‘state of the art’ materials and technologies, (such as smart multifunctional alloys) for CF applications. This could help facilitate development of efficient, cost-effective transducer, actuator, smart structure, vibration dampening and energy harvesting materials for use by the Canadian Forces.

Future work: The unique process for the production of Fe-Ga feedstock materials and the fabrication of textured magnetostrictive Fe-Ga wires has attracted interest from industry and academia. Further studies are warranted to refine the fabrication approach to produce longer and more consistent/uniform diameter wires that poses magnetostriction values > 100ppm. This would be a significant achievement and will widen the design possibilities for future transducers and sensors – especially in blast-prone environments.

Sommaire

Development of Magnetic Actuator Materials for Energy Harvesting: Final Report

R.A. Dunlap, T.D. Hatchard, J.S. Thorne, J.M. Gaudet, S.E. Flynn and M. Maillet; DRDC Atlantic CR 2009-117; R & D pour la défense Canada – Atlantique; septembre 2012

Introduction : Les alliages magnétostrictifs présentent un couplage dynamique des propriétés magnétiques et mécaniques. Le phénomène de couplage en question permet de transformer l'énergie cinétique en énergie magnétique pouvant être récupérée pour produire, dans des conditions adéquates, de l'énergie électrique (et pour exécuter le processus inverse). Les alliages magnétostrictifs pourraient donc se révéler très utiles dans la mise au point des matériaux efficaces et peu coûteux qui servent à fabriquer des transducteurs, des actionneurs, des structures intelligentes et des dispositifs d'amortissement des vibrations et de récupération d'énergie ayant des applications militaires. Le Laboratoire du chantier naval (Atlantique) a entrepris, dans le cadre d'un projet triennal du Fonds d'investissement technologique, des travaux de recherche et développement visant à mettre au point des alliages magnétostrictifs utilisés pour fabriquer des dispositifs de récupération d'énergie.

Un contrat a été conclu avec M. Dunlap (Ph.D.), du département de physique et des sciences de l'atmosphère de l'Université Dalhousie, en vertu duquel des travaux ont été exécutés afin d'évaluer diverses méthodes de fabrication, de traitement et de production d'alliages magnétiques actifs et les propriétés thermiques, magnétiques et cristallographiques des produits obtenus. Un rapport de contrat de recherche précédent (RDDC Atlantique CR 2007-336; janvier 2008) contenait un résumé des principaux résultats obtenus au cours de la première année du projet, lesquels ont servi à établir clairement le plan de recherche exécuté au cours des deux autres années du projet.

Résultats : Le présent rapport contient les résultats importants obtenus au cours des deux dernières années du projet, plus particulièrement ceux ayant trait aux travaux de mise au point d'alliages de Fe-Ga magnétostrictifs. Les résultats démontrent que l'alliage mécanique (par broyage à boulets) et la trempe instantanée (coulage de bandes) constituent des méthodes rentables de production d'alliages de Fe-Ga de diverses compositions. La caractérisation poussée des structures magnétiques et cristallographiques des produits indique qu'ils possèdent des propriétés exceptionnelles. On a élaboré une méthode de fabrication très rentable de tiges brutes en Fe-Ga, lesquelles constituent d'excellentes matières premières pour la production de fils de Fe-Ga magnétostrictifs présentant une texture cristallographique <100>.

Portée : Maintenant que les travaux de recherche du projet sont achevés, leurs résultats ont permis à RDDC de redéfinir clairement son rôle de leader en recherche et d'expert-conseil dans les domaines de l'élaboration de nouveaux matériaux et de l'évaluation de leurs propriétés, ainsi que de l'état de préparation technique et de la pertinence de nouvelles technologies « de pointe » (par exemple le secteur des alliages multifonctionnels) pouvant avoir des applications d'intérêt pour les Forces canadiennes (FC). Cette conjoncture pourrait faciliter la mise au point des matériaux efficaces et peu coûteux qui servent à fabriquer des transducteurs, des actionneurs, des

structures intelligentes et des dispositifs d’amortissement des vibrations et de récupération d’énergie pouvant être utiles aux FC.

Recherches futures : L’industrie et le milieu universitaire ont démontré un vif intérêt pour le procédé exceptionnel qui permet de produire des matières premières à base d’alliages de Fe-Ga et de fabriquer des fils de Fe-Ga magnétostrictifs ayant une texture cristallographique déterminée. L’exécution d’études plus poussées est justifiée, car il est important de perfectionner la méthode de fabrication de fils plus longs et de diamètre plus uniforme, qui présentent des valeurs de magnétostriction mesurables correspondant à des concentrations supérieures à 100 ppm. Ce serait là une réalisation importante qui permettrait d’élargir le domaine des futurs modèles possibles de transducteurs et de capteurs, particulièrement ceux utilisés dans des milieux où peuvent se produire des explosions.

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Acknowledgements

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1 Introduction

Magnetostrictive and magnetic shape memory alloys are smart magnetically-active materials that exhibit dynamic coupling of magnetic and mechanical properties. These alloys are attractive for applications where transforming kinetic energy to magnetic energy to electrical energy is warranted. While energy harvesting was the focus of the current research, other applications include sensors, actuators, transducers, adaptive structures and vibration dampening systems.

The application of these, and similar materials, relies upon the engineering of materials with appropriate physical properties such as structural and magnetic transition temperature, saturation magnetization, magnetic anisotropy energy and stress-strain relations. To this end, the Dockyard Laboratory (Atlantic) undertook a 3 year Technology Investment Fund project to research and develop magnetostrictive alloys for energy harvesting applications.

Dr. Dunlap of Dalhousie University (Department of Physics and Atmospheric Science) was contracted to evaluate fabrication and processing approaches for production of magnetically active alloys and to evaluate the resultant thermal, magnetic and crystallographic properties. This study was contracted over 3 years. The first year examined a variety of techniques for fabrication of Ni-Mn-Ga magnetic shape memory alloys and magnetostrictive Fe-Ga alloys but focused on mechanical alloying (ball-milling) [1]. This had helped define the research plan for the remaining 2 years.

The final two years focused primarily on fabrication of Fe-Ga alloys by melt spinning (ribbons) and suction casting (rods). Results are documented in this report. The effectiveness of different zone melting techniques and heat treatments on the grain structure and crystallographic texture of suction extracted rods had been investigated. The magnetostriction was measured and was related to the texture. A manuscript describing the results of this phase of the work was published in an international journal [2]. A copy of the manuscript was included (Annex A) to provide more comprehensive information.

This document is organized as follows. In Section 2, relevant background information is presented. Methods for the production and characterization of suction cast FeGa rods will be discussed in Section 3 and 4, respectively. Conclusions are presented in Section 5. Section 6 provides an overall summary of the conclusions of 3 phases of the 3-year project as well as future work. Annex A contains a manuscript, while Annex B lists publications and presentations that have resulted both directly and indirectly (co-authored) from this 3 year contract.

2 Background

The magnetostriction of Fe can be greatly increased by the addition of Ga [2-8]. Alloys of the form $Fe_{100-x}Ga_x$ with x around 19 at% have attracted interest because of their large magnetostriction and have been considered as possible candidates in place of the more traditional rare-earth containing alloys for applications as sensor or actuator materials. It is known that there is a close correlation between the structure of these alloys and their resulting magnetoelastic properties. In particular, it is commonly believed that Ga-Ga pairing in alloys near $x = 19$ is responsible for an increase in magnetostriction and that the formation of an ordered $D0_3$ phase as the alloy composition approaches $x = 25$ is detrimental to this property [9]. Recent reports have shown that the microstructure of these alloys may be influenced by processing conditions and that rapid quenching from the melt [10-12], combinatorial sputtering [13] and mechanical alloying methods [14] may provide a means of controlling the microstructure and hence the magnetostriction.

Besides the composition, chemical ordering and microstructure, the overall macroscopic structure of a final product is important. While single crystal samples should provide the highest magnetostriction for an alloy of a given composition, the cost of producing and processing single crystals is very high in terms of both dollars and time. Also, the complexity of single crystal production may be a barrier to mass production. In order to avoid the drawbacks of single crystals, researchers have turned to studying polycrystalline samples. Polycrystalline samples can be produced much more easily and quickly, and at much less expense. However, in a randomly oriented polycrystalline sample, the direction of maximum magnetostriction for each individual crystallite will not be the same as all the others, and some reduction of the magnetostriction relative to that of single crystals will be encountered. In order to maximize magnetostriction of a polycrystalline sample, efforts have been made to align the crystallites so that the direction of maximum magnetostriction for the crystallites is common throughout the sample. Some recent work has focused on making oriented Fe-Ga rods through stress annealing [15] or rapid quenching from an under-cooled melt [16] and oriented Fe-Ga wires by drawing using a modified Taylor wire method [17].

3 Experimental Methods

Following previous investigations of melt spun and mechanically alloyed Fe-Ga [1, 10-12. 14], methods of preparing bulk Fe-Ga were considered. Investigations have focused on the suction casting technique as this should provide samples of a suitable geometry for magnetostrictive sensors and actuators. Zone annealing and zone melting were being investigated for their effectiveness for grain growth and texture development. The magnetostrictive properties of rods formed in this way may be investigated using traditional strain gauge techniques.

3.1 Preparation of rods

Fe-Ga rods also hold promise as precursor materials for formation of Fe-Ga wires using the Taylor wire method. The suction extraction technique follows from that developed by Srinivas and Dunlap [18] and discussed in more detail in Hatchard et al. [2]. The apparatus constructed for this investigation is illustrated schematically in Figure 1. Figure 2 shows a photograph of the apparatus.

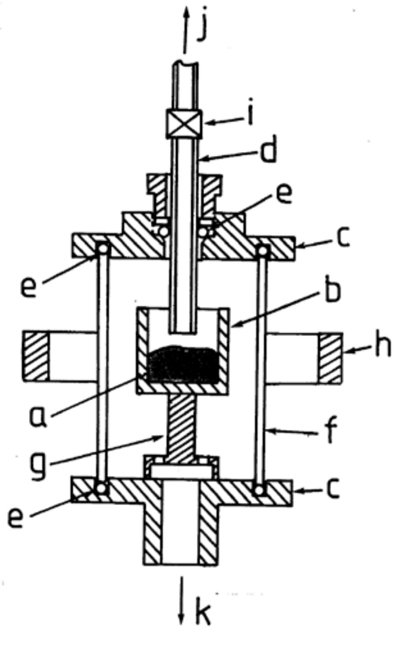

Figure 1: *Schematic of the suction casting apparatus; (a) Fe-Ga sample, (b) quartz crucible, (c) vacuum flange, (d) quartz suction extraction tube, (e) vacuum seal, (f) outer quartz tube, (g) support, (h) induction coil, (i) valve, (j) to vacuum pump for suction extraction and (k) to vacuum system for evacuation and back-filling.*

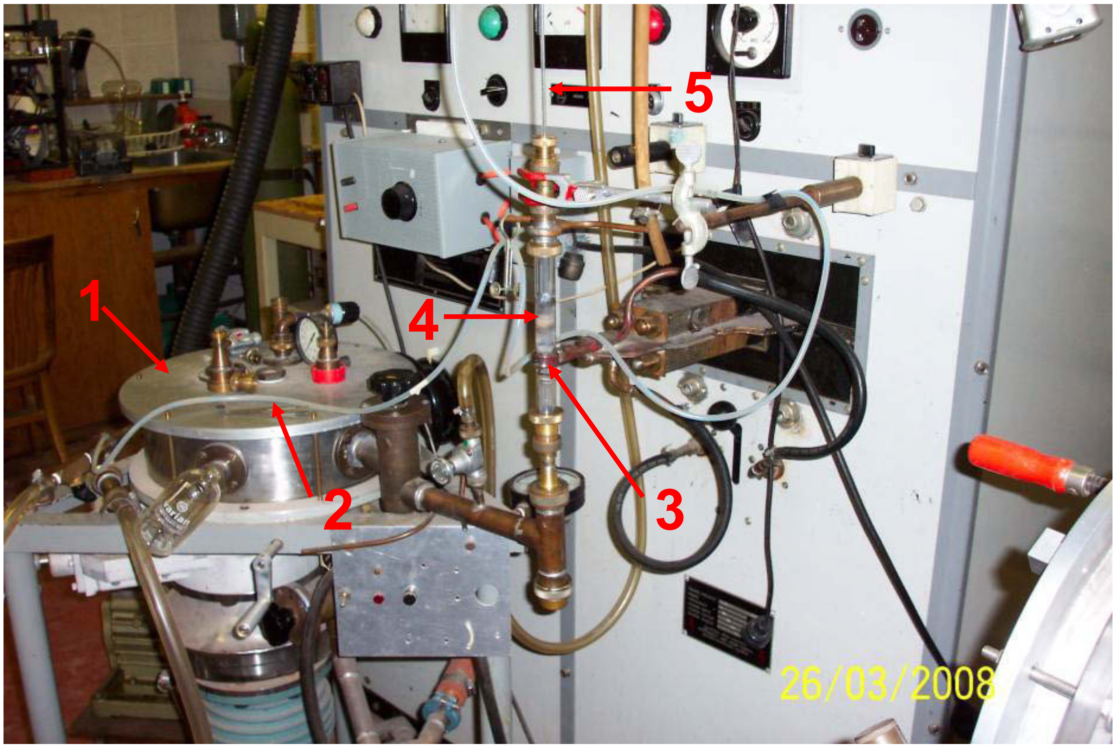

Figure 2: *Photograph of the suction casting apparatus; (1) vacuum pump, (2) plastic suction hose, (3) induction coil, (4) outer quartz tube and (5) quartz suction tube.*

Rods were prepared from Fe (Johnson Matthey, 99.98%) and Ga (Alfa Aesar, 99.999%) with a starting composition of approximately $Fe_{79.5}Ga_{20.5}$ to target a final composition of $Fe_{81}Ga_{19}$. Excess Ga was used in the precursor material to account for a Ga loss of 1 to 2 atomic percent during induction melting as observed in previous work [12]. Once the precursor material is molten, a quartz tube is lowered into the melt and material is drawn into the tube where it rapidly solidifies using a rotary vacuum pump. The rods of Fe-Ga formed in this way are referred to as as-cast rods. Typical rod diameters are from 1 to 3 mm, depending on the inner diameter of the quartz tube used to extract the melt from the crucible.

Fe-Ga rods up to about 28 cm in length have been prepared by this method. A photograph of two different diameter rods (1 mm and 3 mm diameter) is shown in Figure 3. As-cast rods were subsequently sealed under an Ar atmosphere in quartz tubes for zone melting. Zone melting was performed with the rod, sealed in quartz, suspended vertically. A H_2/O_2 torch was used to re-melt the rod. The rod was heated at the bottom until it became molten and then the torch was moved up the rod, holding at one spot long enough to melt the material before proceeding. With this method about one cm of rod was molten at a given time. The entire procedure lasted about 10 to 15 minutes for a 20 cm rod. After the zone melting, some rods were subsequently annealed under flowing Ar for 4 hours at 650 °C.

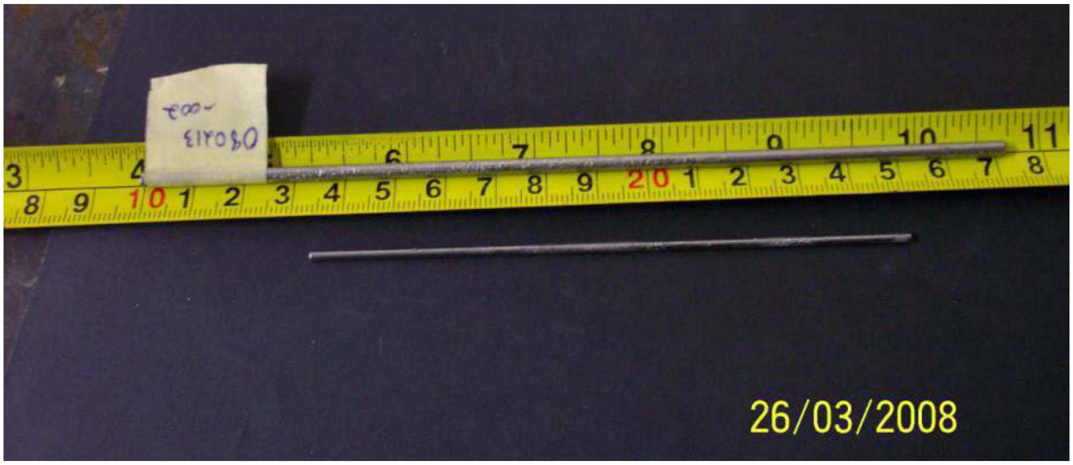

Figure 3: *Fe-Ga suction cast rods. The smaller rod is about 1.9 mm in diameter and 12 cm long and the larger rod is about 3.2 mm in diameter and 17 cm long.*

Compositional analysis was performed with a JEOL 8200 microprobe using wavelength dispersive spectroscopy (WDS). X-ray diffraction (XRD) patterns were obtained at room temperature for all samples using Cu-K_α radiation with a Siemens D-500 scanning diffractometer. Magnetization measurements were obtained at room temperature using a PAR 155 vibrating sample magnetometer (VSM). Electron backscatter diffraction was performed using a Channel 5 EBSD camera and software from HKL Technology on a Cold Field Emission Hitachi S-4700 FEG SEM (operated at 20 kV and 30 mA). Radial and axial cross sections of the rods were cut and polished to a 0.05 micron finish for EBSD. The radial sections were also used for x-ray diffraction. Magnetostriction was measured by capacitance dilatometry at St. Francis Xavier University.

3.2 Heat treatment of rods

3.2.1 Zone annealing

The apparatus for zone annealing is illustrated in Figure 4. The sample was contained in an inner tube and was held fixed while the furnace, which is suspended by cables and balanced by counterweights, was translated up or down. The translation rate was controlled by the computer system shown to the right in Figure 4. Figure 5 shows the quartz tube that houses the furnace and the details of the heating element are shown in Figure 6.

Initial tests produced zone annealed rods that were characterized by the methods discussed in section 4. Results were compared to results from other heat treatment methods and are discussed in Section 4. Results will be compared to results from other heat treatment methods to determine the best approach for modifying the crystallographic texture in the rods.

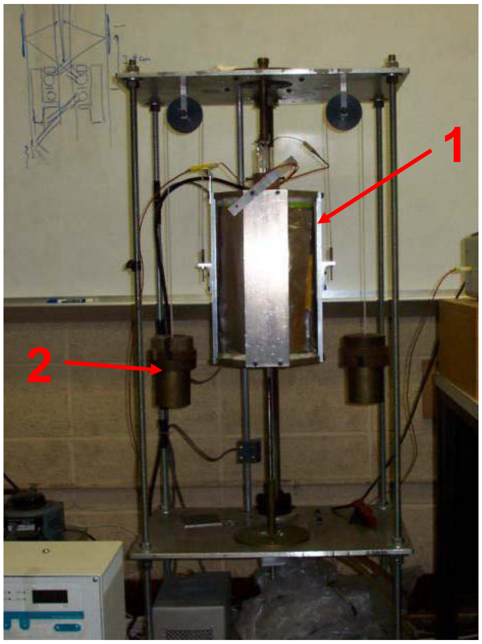

Figure 4: *Zone annealing system; (1) furnace and (2) counter weight.*

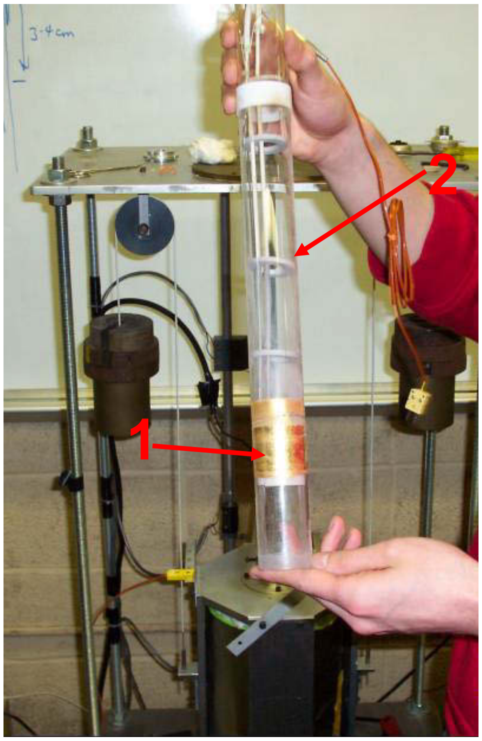

Figure 5: *The inside of the zone annealing furnace; (1) quartz tube and (2) heater.*

Figure 6: *Zone annealing furnace heater showing heating coils.*

3.2.2 Zone melting

The zone melting apparatus is shown in Figure 7. The sample, contained in a slightly oversized quartz tube, was zone heated to melting by the induction furnace. The induction coil arrangement is illustrated in Figure 8. The sample was attached to either the upper or lower translation rod and pulled or pushed through the heating zone at a controlled rate. The translation rod on the other end was used as a guide to keep the Fe-Ga rod in a vertical orientation. Initial zone melting tests have been conducted and the effect of zone melting on grain growth and texture development in Fe-Ga rods are presented in Section 4.

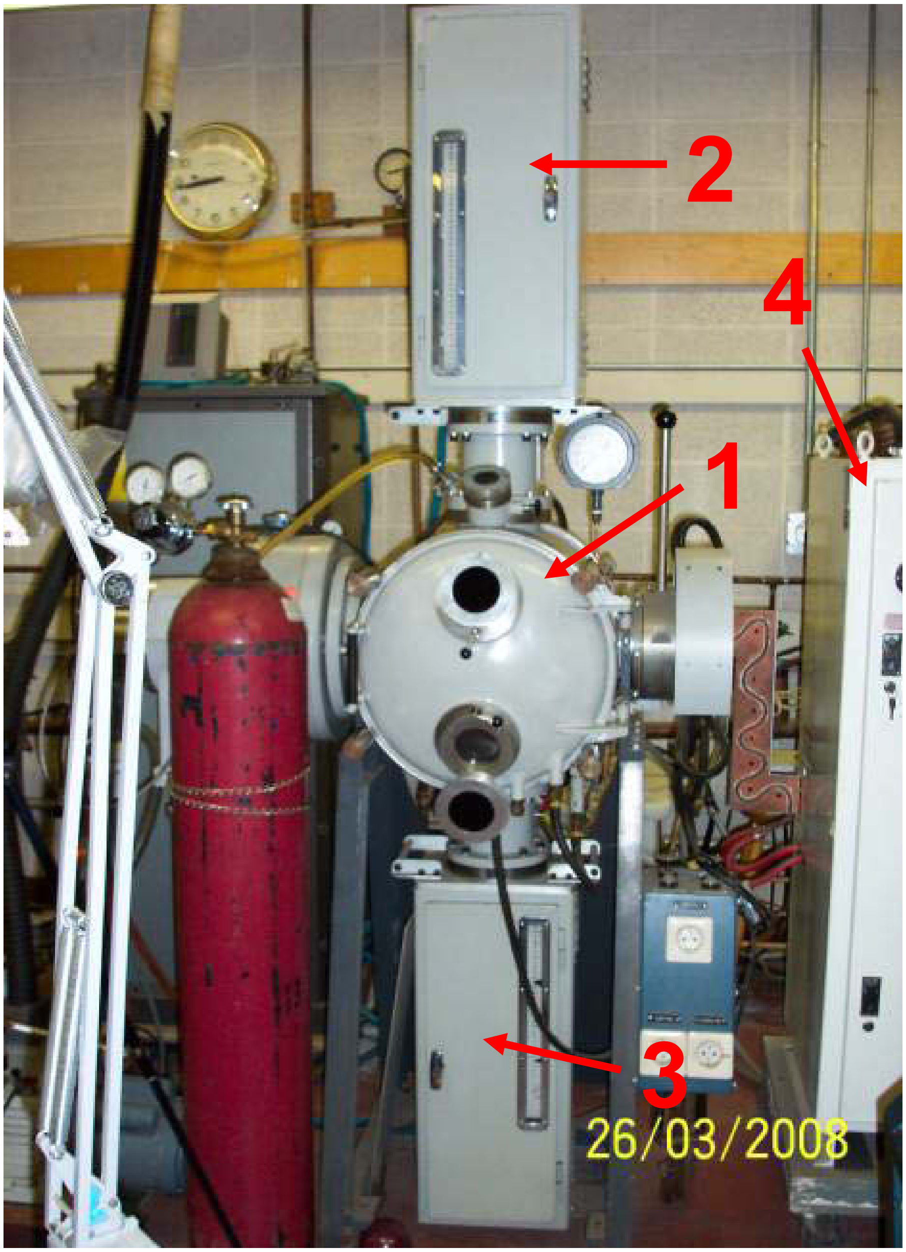

Figure 7: *Zone melting system; (1) vacuum chamber, (2) upper translation stage, (3) lower translation stage and (4) induction generator.*

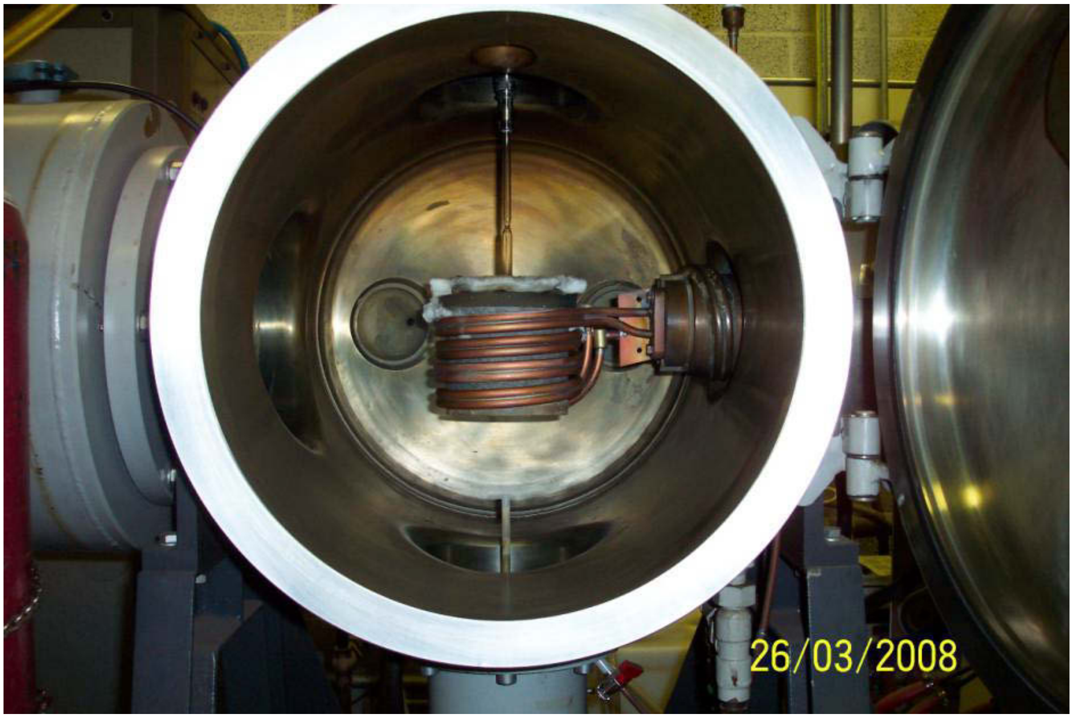

Figure 8: *Interior of zone melting vacuum chamber showing induction coil and upper and lower translation rods.*

Figure 9: *Interior of induction coil showing graphite susceptor.*

4 Results and Discussion

4.1 As-cast rods

The X-ray diffraction pattern collected from a radial cross section of a 3 mm diameter as-cast rod is shown in Figure 10a. The pattern is essentially that of a single phase bcc structure with the relative peak intensities indicating a randomly oriented polycrystalline sample. The magnetization versus applied field for this sample is shown in Figure 11. The field was applied parallel to the axis of the rod using a 4 mm long section. Results indicated that the sample is magnetically quite soft with a narrow hysteresis loop. Saturation occurs at an applied field of just over 4 kG. The saturation magnetization was measured to be 200 emu/g. This was nearly 15 % higher than the expected value of ~175 emu/g. The saturation magnetization of Fe-Ga alloys was estimated by multiplying the saturation magnetization of pure Fe (218 emu/g) by the composition of ferromagnetic Fe (measured as 80 % Fe by mass using WDS).

Figure 11 shows the magnetostriction of the as cast sample in parts-per-million (ppm) versus applied field. The sample has a maximum magnetostriction of 50 ppm, and does not saturate until the applied field reaches 8 kG. The field is again applied parallel to the length of the rod using a 4 mm long section. There is also some hysteresis in the measurement, with a remnant magnetostriction of about 10 ppm. The magnetostriction measured here is only about 20 % of values reported for single crystals of Fe-Ga with similar compositions when measured along the <100> direction [3, 4, 19, 20].

Figure 13a shows the results of electron backscatter diffraction (EBSD) measurements performed on the cross section of the as-cast sample. Figure 13a shows the orientation map for this sample with the color code shown in Figure 13b. Selected pole figures are shown in Figure 13c. It is clear that the sample is made up of many randomly oriented grains a few 10's of microns in diameter. This helps to explain why the magnetostriction for this sample is so low in comparison to a single crystal. In the as-cast sample, very few of the grains have the <100> direction aligned along the axis of the rod. This means that for most of the sample, the field has not been applied along the direction of maximum magnetostriction while performing the dilatometer measurements. For a polycrystalline sample to produce large magnetostriction it therefore must have a preferred orientation.

4.2 Zone-melted rods

The X-ray diffraction pattern collected from a radial cross section of a 3 mm diameter zone-melted rod is shown in Figure 10b. The pattern collected for this rod is different from the as-cast rod. All of the peaks for bcc-Fe are present, but the ratio of peak intensities is no longer consistent with a randomly oriented sample. The (110) and (211) peaks have nearly disappeared, while the (200) peak (the weakest peak in the diffraction pattern of the as-cast material) dominates the pattern. This is an indication that the planes parallel with the sample surface are predominantly {200} and that the sample therefore has a preferred <100> orientation along the axis of the rod.

This <100> orientation is confirmed by the EBSD data for the zone-melted sample, which is shown in Figure 14. The orientation map in Figure 14a shows that the rod (at the location of this cross section) is predominantly one large grain which has the {100} planes parallel to the surface. There are a few smaller grains around one side, one of which does and one does not have {100} planes parallel to the surface. The one grain that is significantly different than the others is still only about 10° from the <100> direction. The (100) pole figure for this sample in Figure 14c also is indicative of a sample with a preferred <100> orientation. The cluster of data points near ½ the radius in the (110) and ⅔ the radius in the (111) pole figures indicate a clustering of crystallographic planes that are nearly 45 and 60 degrees from (110) and (111), respectively. This is consistent with the {100} planes. The clustering indicates that the crystallographic directions are rotated to some extent from one another around the axis of the rod (the z-axis for the pole figures).

The zone melting process had little to no effect on the magnetization curve for the sample (Figure 11b). The shape and saturation magnetization are unaffected. To verify that the grain growth and orientation of the zone-melted sample were not localized to the cross section (Figure 14a), an axial section was polished and EBSD data were collected. The resulting orientation map is shown in Figure 15, using the same colour code as the two previous maps. The section measured was approximately 1 cm in length, and found to be essentially one grain at the polished surface with an orientation about 10° from <100> (the orientation would have to be from 0 – 45° from <100> based on Figure 14b and the cubic structure of the sample). The combination of the XRD and EBSD data strongly indicate that the zone-melted rod is made up of quite large grains with the <100> direction aligned very near to the long axis of the rod.

Application of a magnetic field parallel to the rod axis will also be parallel with the <100> direction of maximum magnetization of Fe-Ga alloys. The magnetostriction of the zone-melted rod was also measured with a capacitance dilatometer. The results are shown in Figure 16. As can be seen, the magnetostriction of the rod has been doubled by the zone-melting process to 100 ppm. The overall shape of the curve is still the same, with saturation occurring above 4 kG and with slightly less hysteresis. While the magnetostriction of the zone-melted rod is still somewhat less than maximum values reported for single crystals of similar composition, the techniques used to produce and process the rod are much faster, less complex and less expensive than techniques used to obtain single crystals. Also, the composition of this rod as measured by WDS is $Fe_{82.4}Ga_{17.6}$. The magnetostriction is expected to increase with Ga content up to about 19%, so there may be room to increase the magnetostriction by adding more Ga to the precursor mix used to cast the rod.

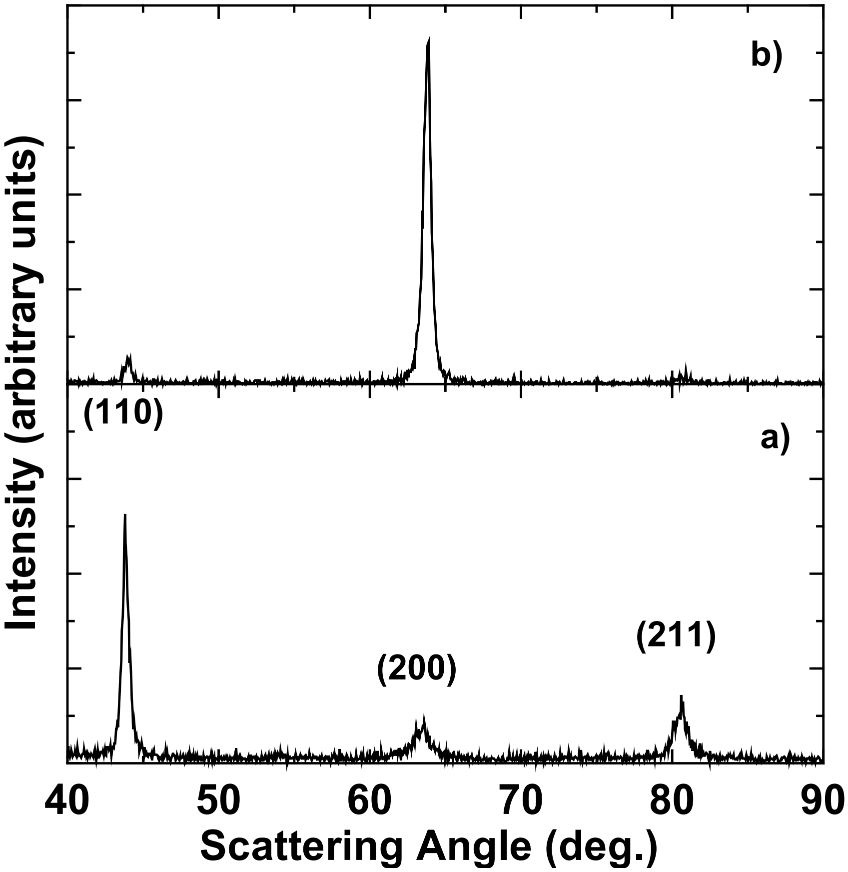

Figure 10: *X-ray diffraction patterns of radial cross sections of (a) an as-cast rod and (b) rod that has been zone melted.*

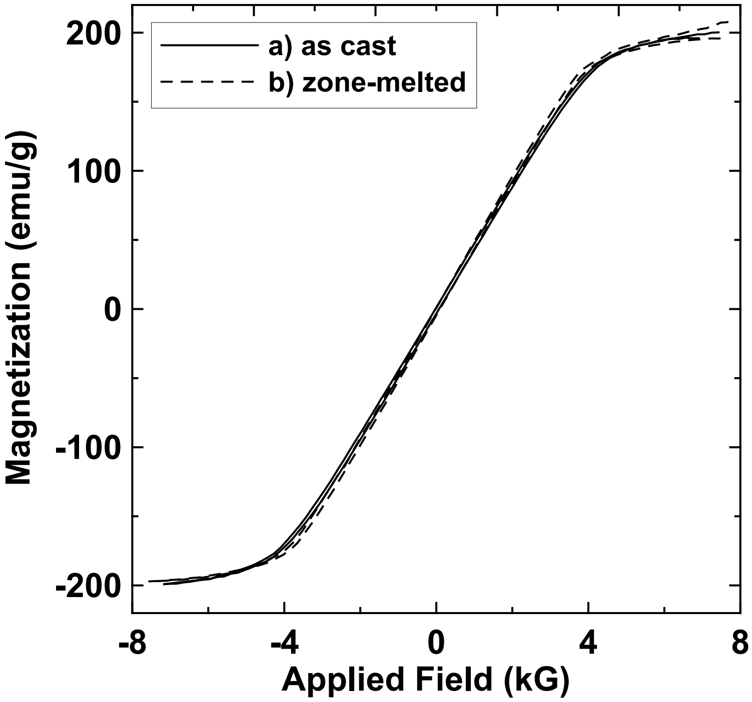

Figure 11: *Magnetization versus applied field for (a) as cast rod and (b) zone-melted rod.*

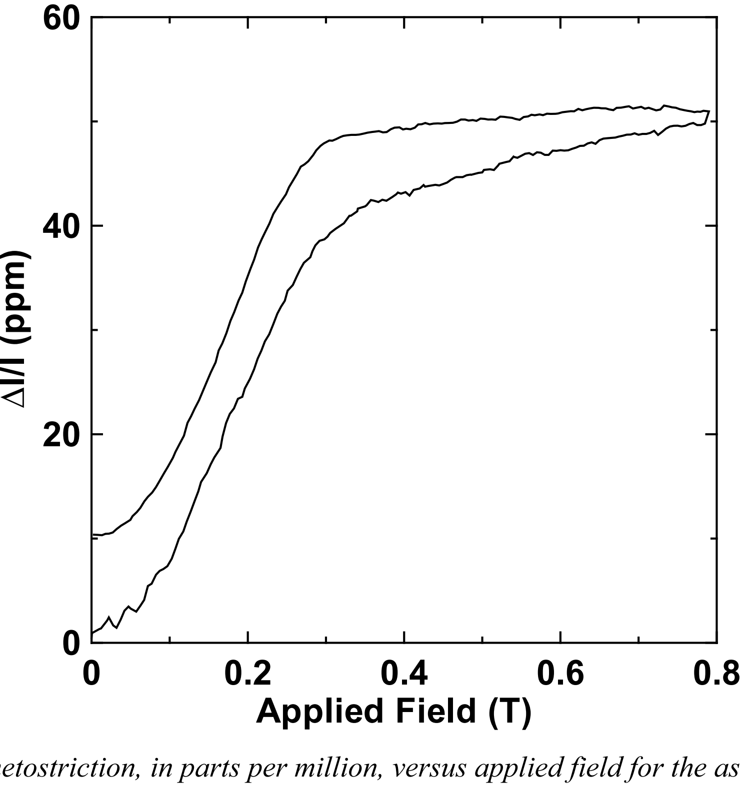

Figure 12: *Magnetostriction, in parts per million, versus applied field for the as cast Fe-Ga rod.*

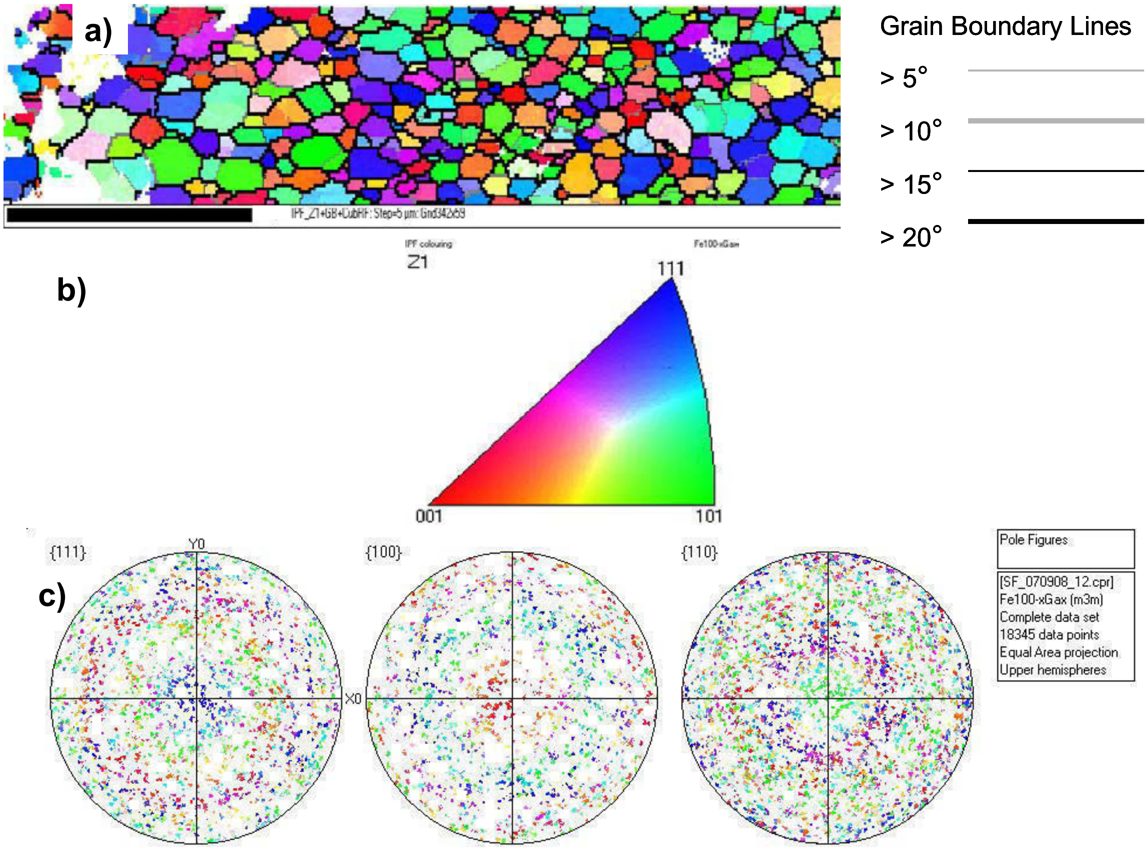

Figure 13: *EBSD data for a radial cross section of the as cast rod showing (a) orientation map, (b) color code and (c) selected pole figures. The scale bar in a) is 700 µm.*

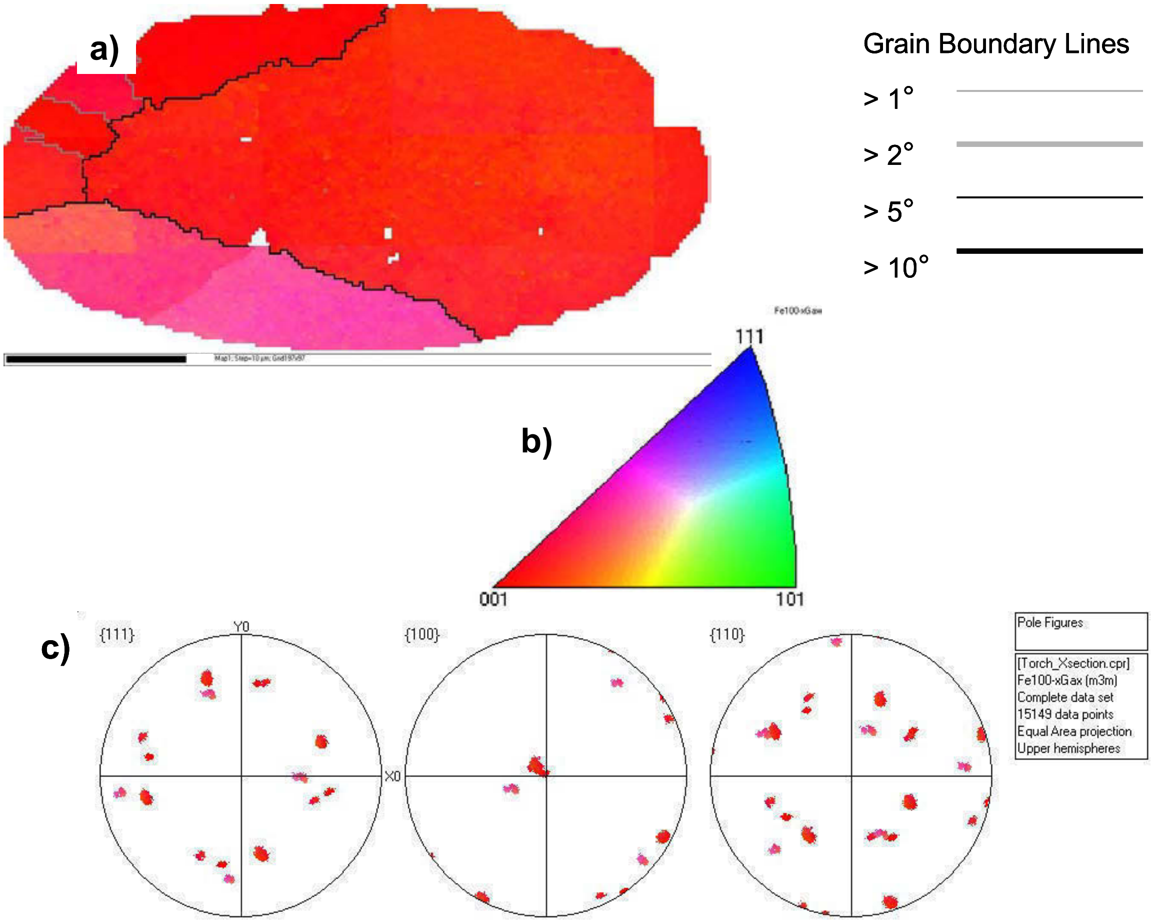

Figure 14: *EBSD data for a radial cross section of the zone melted rod showing (a) orientation map, (b) color code and (c) selected pole figures. The scale bar in (a) is 700 µm.*

Figure 15: *Orientation map for an axial cross section of the zone melted using the same colour code and grain boundary markers as Figure 14. The scale bar is 3000 µm.*

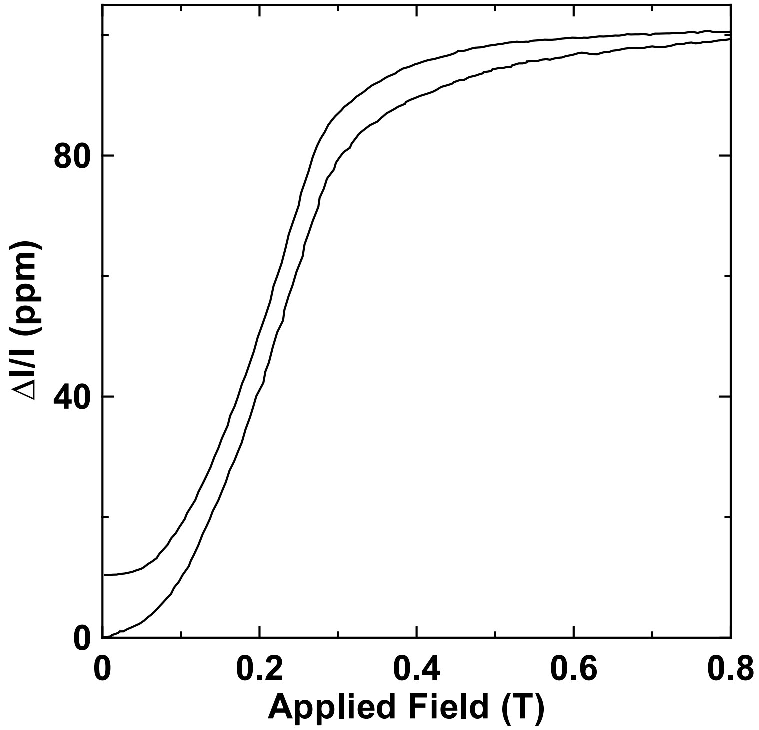

Figure 16: *Magnetostriction, in parts per million, versus applied field for the zone-melted Fe-Ga rod.*

4.3 Annealed zone-melted rods

In an effort to increase the magnetostriction of zone-melted rods, portions of the rods were annealed under flowing Ar at a temperature of 650 °C for 4 hours. It was hoped that the annealing process would allow grain growth and improve texture of the crystallites. The XRD, VSM and EBSD data for annealed zone-melted rods are all very similar to the zone-melted rods that were not annealed. The composition as measured by WDS remained unchanged.

The magnetostriction was significantly affected by the annealing. As can be seen from Figure 17, the magnetostriction for the annealed zone-melted rod has been reduced from 100 ppm down to 25 ppm. The reasons for the decrease in the magnetostriction of the annealed rod can readily be seen from the EBSD measurements of a radial cross section as shown in Figure 18. The grains have reoriented to an approximately <111> texture from the preferred <100> crystallite texture seen in the zone melted rod. A measurable decrease in grain size is also seen indicating that some minor mis-orientation of <100> crystallites in the zone melted rod have adopted more significantly different orientations on annealing. This reorientation may be the result of surface effects.

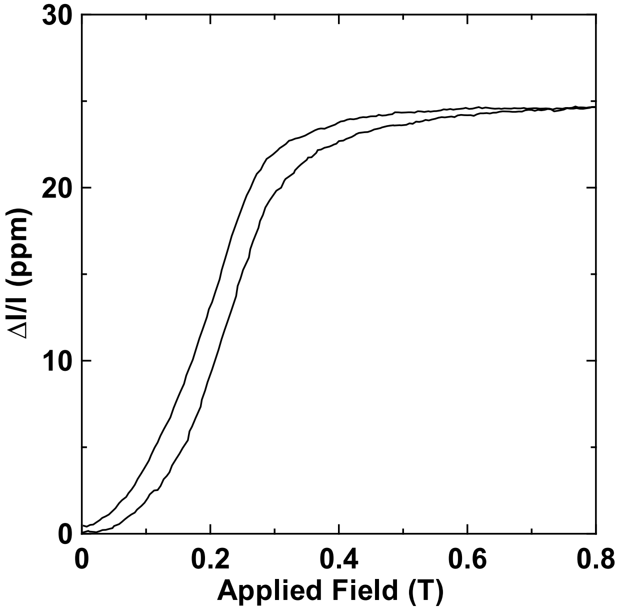

Figure 17: *Magnetostriction versus applied field for an annealed zone-melted Fe-Ga rod.*

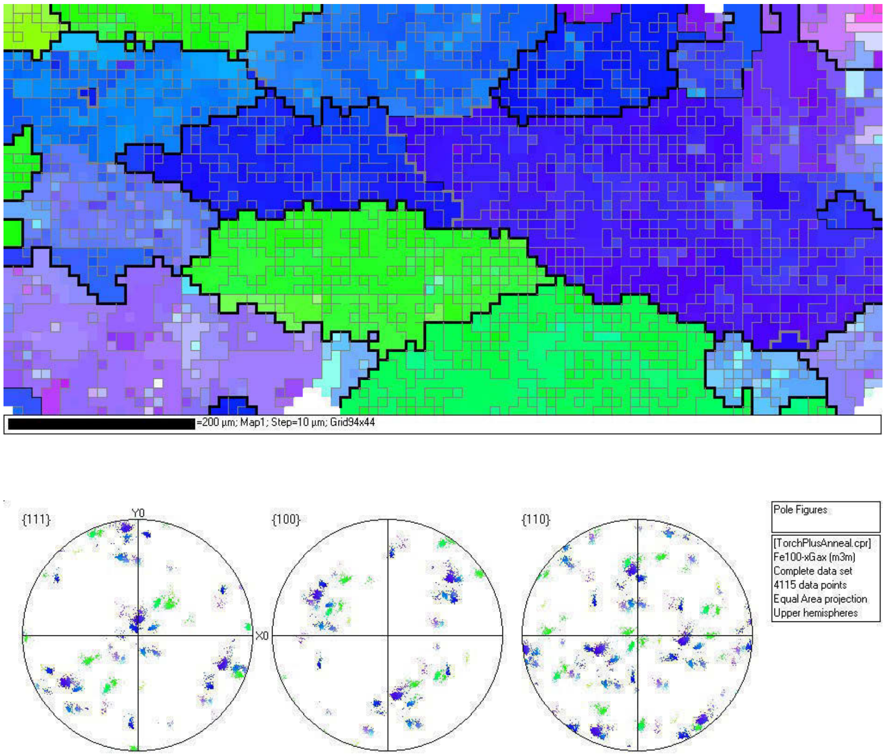

Figure 18: *EBSD map (top) and pole plots (bottom) for a radial cross section of a rod that has been zone melted and subsequently annealed. This figure uses the same color scale that was shown in Figure 14b.*

5 Conclusions

The production and characterization of predominantly <100> oriented magnetostrictive Fe-Ga rods have been described. Polycrystalline Fe-Ga rods, 1-3 mm in diameter and up to 20 cm in length were rapidly produced using the suction extraction technique. Post fabrication processing approaches were employed to improve texture and magnetostriction of the Fe-Ga rod precursor. In this work, the maximum Ga content in a fully processed rod was less than 18 atomic percent. The Ga loss between the elemental precursor and the suction extracted rods was about 2 – 4 atomic percent, larger than the 1 – 2 atomic percent loss experienced for ball milling [14] or melt-spinning [12]. This leaves the door open for future optimization of the final rod composition.

It was found that the zone-melting process is the preferable post-fabrication processing method as it leads to higher magnetostriction. Zone-melting was shown to increase grain size, improve the <100> texture and produce large magnetostriction values (up to 100 ppm) in the final product. Subsequent annealing reoriented the crystallite grains into a preferential <111> texture leading to a significant reduction in the magnetostriction (near 25 ppm).

The highest magnetostriction for Fe-Ga alloys in the literature seems to be found at slightly higher Ga contents than rods studied here [3-9]. It has also been shown that the maximum Ga content achieved before the onset of $D0_3$ ordering is dependent on the processing technique(s) used [10-14]. A further study is needed to find the optimal final composition for a zone-melted rod in order to maximize magnetostriction.

6 Summary of the 3 Phases and Future Work

Work conducted during the three years of the TIF project dealt with two different classes of materials for potential energy harvesting applications. Ferromagnetic shape memory alloys of the Ni-Mn-Ga system were studies during years 1 and 2 and traditional magnetostrictive alloys based on Fe-Ga were studied primarily during years 2 and 3.

The Ni-Mn-Ga studies followed from previous collaborative work (prior to this contract) with DRDC and considered two aspects of these materials; the understanding of the influence of composition and structure on the magnetic interactions and the methods for the production of suitable materials for making alloy/polymer composites. During phase 1, work on the preparation of Ni-Mn-Ga powders by ball milling has made significant contributions in both these areas. The effects of chemical ordering on magnetic properties have been understood in the context of Mn-Mn indirect exchange coupling. Magnetic shape memory alloy powders produced by ball milling have been used in preliminary investigations at DRDC of alloy/polymer composite preparation. Further studies of magnetic shape memory alloys should include more detailed analyses of the effects of composition on magnetic properties and the application of theoretical models of indirect exchange interactions for the optimization of materials properties. Investigations of polymer composites should include the careful consideration of polymer properties in order to optimize coupling of the mechanical properties of the polymer and the magnetoelastic properties of the shape memory alloy. Further development of composites would require the optimization of particle size, loading factor as well as the magnetic orientation of grains during polymer solidification.

The studies of magnetostrictive Fe-Ga alloys included the investigation of mechanically alloyed powders (ball-milled; phase 1 and 2), rapidly solidified ribbons (melt spinning; phase 1) and zone melt rods (suction casting; phase 2 and 3). Properties of mechanically alloyed powders and rapidly solidified ribbons have provided insight into the formation of Fe-Ga phases and their composition dependence. This also included the effects of sample composition and preparation methods on microstructure and magnetic and crystallographic structure (particularly chemical ordering). These factors are of direct significance in determining the resulting magnetostriction. Further studies of the application of Fe-Ga/polymer composites for energy harvesting would follow along the lines described above for mechanically alloyed shape memory alloy powders.

Studies of suction extracted rods have provided the best approach to obtaining technically useful materials with optimized magnetostrictive properties. Suction extraction provides rods of suitable dimensions for use as bulk materials or as convenient precursors for wire production. Zone melting studies have shown that directional solidification is an effective method for the production of a desirable <100> axial texture in the rods. Further studies have shown that this desirable texture is eliminated by annealing.

Further studies of the production of oriented rods should include the optimization of sample composition and zone annealing parameters and a more thorough understanding of the relationship of these properties and the resulting magnetostriction. The application of backscatter Mossbauer spectroscopy would further elucidate the influence of zone melting methods on chemical order and in particular Ga ordering in the bcc Fe lattice. The ability to perform

Mossbauer and magnetostriction studies on the same bulk samples will be invaluable in clarifying the effects of Ga order on magnetostriction.

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Annex A Manuscript, “Production and characterization of <100> textured magnetostrictive Fe-Ga rods”

“Production and characterization of <100> textured magnetostrictive Fe-Ga rods”

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Abstract

Magnetostrictive rods of the approximate composition $Fe_{82}Ga_{18}$ have been produced with a preferred <100> axial crystallographic texture. Randomly oriented polycrystalline rods were cast by a suction extraction technique and a preferred <100> axial crystallographic texture was introduced through zone melting. Capacitance dilatometery showed that the as-cast rods and oriented zone-melted rods had saturation magnetostrictions of approximately 50 ppm and 100 ppm, respectively. Subsequent annealing of zone melted rods resulted in a re-orientation of the axial crystallographic texture to a preferential <111> direction along with a corresponding reduction of the saturation magnetostriction.

***Keywords**:* Magnetostrictive materials, Fe-Ga, Ferromagnetic alloys, Crystallographic texture, Suction extraction, Zone melting, X-ray diffraction

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1. Introduction

The magnetostriction of Fe can be greatly increased by the addition of non-magnetic elements such as Ga [1-6]. $Fe_{100-x}Ga_x$ alloys with x around 19 at% have attracted interest because of their large magnetostriction and have been considered as possible candidates to replace the more traditional rare-earth-containing magnetostrictive alloys for applications as sensor or actuator materials. It is known that there is a close correlation between the structure of these alloys and their resulting magnetoelastic properties [7]. In particular, it is commonly believed that Ga-Ga pairing which is a maximum in alloys with Ga content around 19 at% is responsible for an increase in magnetostriction and that the formation of an ordered $D0_3$ phase for higher Ga content is detrimental to this property [7]. Recent reports have shown that the microstructure of these alloys may be influenced by processing conditions and that rapid quenching from the melt [8-10], combinatorial sputtering [11] and mechanical alloying methods [12] may suppress the formation of the $D0_3$ phase and may, therefore, be a mechanism for controlling the magnetostriction.

Besides the composition and chemical ordering, crystallographic orientation plays an important role in determining the magnetostriction where the maximum value is obtained along the <100> direction. Thus while single crystal samples should provide the highest magnetostriction for an alloy of a given composition, such samples are expensive and time consuming to produce. Also, the complexity of single crystal production may be a barrier to mass production. In order to avoid the drawbacks of single crystals, researchers have turned to studying polycrystalline samples that can be produced much more easily and economically [e.g. 13]. However, polycrystalline alloys suffer from a reduction in the maximum achievable magnetostriction over their single crystal counterparts. This is because the direction of the maximum magnetostriction (<100> for Fe-Ga) in a randomly oriented polycrystalline sample will not be the same for all crystallites. In order to achieve higher magnetostriction from a polycrystalline sample, a variety of processing approaches have been investigated. Some recent work has focused on the stress annealing of Fe-Ga rods [13], rapid quenching from an under-cooled melt [14], the production of crystallographically oriented Fe-Ga wires using a modified Taylor wire method [15] and the zone melting of arc melted Fe-Ga rods [16].

In the present work, a method for casting polycrystalline Fe-Ga rods and then inducing axial <100> crystallographic texture by zone melting using rates that are substantially greater than those reported previously is described. The effects of annealing zone melted rods were also investigated. The structural and magnetostrictive properties of both as-cast, zone-melted and annealed rods are presented.

2. Experimental methods

Rods of Fe-Ga were prepared using the suction extraction technique as described in [17]. Pieces of Fe (Johnson Matthey, 99.98%) and Ga (Alfa Aesar, 99.999%) were placed in a quartz crucible and melted by induction heating. The starting material had an overall composition of approximately $Fe_{79.5}Ga_{20.5}$ with a final target composition of around $Fe_{81}Ga_{19}$. Excess Ga was used in the starting material to account for a Ga loss of 1 – 2 atomic percent during induction melting as observed in previous work [10]. Once the precursor material was molten, a quartz tube was lowered into the melt and material was drawn into the tube using a rotary vacuum pump. The melt solidified in the quartz tube forming a rod, which is subsequently referred to as the as-cast rod. Typical rod diameters were from 1 – 3 mm, depending on the inner diameter of the quartz tube used to extract the melt from the crucible. Fe-Ga rods up to about 28 cm in length have been prepared by this method. A photograph of two different diameter rods (1 mm and 3 mm diameter) is shown in Figure 1. As-cast rods were subsequently sealed under an Ar atmosphere in quartz tubes for zone melting. Zone melting was performed with the rod, sealed in quartz, suspended vertically. A H_2/O_2 torch was used to re-melt the rod. The rod was heated at the bottom until it became molten and then the torch was moved up the rod, holding at one spot long enough to melt the material before proceeding. With this method about one cm of rod was molten at a given time. The entire procedure lasted about 10 minutes for a 20 cm rod giving a zone melting rate of about 1200 mm/hr. This is substantially greater than rates used for conventional Bridgman methods (2 - 4 mm/hr) and greater than rates used in previous rapid zone melting investigations (25 - 350 mm/hr) [17]. After the zone melting, some rods were subsequently annealed under flowing Ar for 4 hours at 650 °C.

Compositional analysis was performed with a JEOL 8200 microprobe using wavelength dispersive spectroscopy (WDS). X-ray diffraction (XRD) patterns were obtained at room temperature for all samples using Cu-K_α radiation with a Siemens D-500 scanning diffractometer. Electron backscatter diffraction was performed using a Channel 5 EBSD camera and software from HKL Technology on a Cold Field Emission Hitachi S-4700 FEG SEM (operated at 20 kV and 30 mA). Radial and axial cross sections of the rods were cut and polished to a 0.05 micron finish for EBSD. The radial sections were also used for x-ray diffraction. Magnetostriction was measured by capacitance dilatometry using methods described previously [18, 19]. Measurements were made parallel to a magnetic field applied along the axial direction of the rod and no pre-stress was applied.

3. Results and discussion

3.1. As-cast rod

The composition of rods described in the present study was approximately $Fe_{82}Ga_{18}$ (as determined by WDS) and showed no measurable radial or axial variations. Typically, slightly more Ga loss was seen than for other Fe-Ga sample preparation methods that have been reported previously [10]. The X-ray diffraction pattern collected from a radial cross section of a 3 mm diameter as-cast rod is shown in Figure 2a. The pattern is essentially that of a single phase bcc structure with no indication of preferred crystallite texture on the basis of the relative diffraction peak intensities. No evidence of $D0_3$ superlattice peaks have been observed for this or any of the heat treated samples.

Figure 3 shows the magnetostriction of the as-cast sample in parts-per-million (ppm) versus applied field. The sample was found to have a saturation magnetostriction of about 50 ppm. The magnetic field was applied parallel to the length of the rod using a 4 mm long section. There is also some hysteresis in the measurement, with a remnant magnetostriction of about 10 ppm. The magnetostriction measured is only about 20 % of values reported for single crystals of Fe-Ga with similar compositions when measured along the <100> direction [1, 2, 20, 21].

Figure 4 shows the results of electron backscatter diffraction (EBSD) measurements performed on a radial cross section of the as-cast sample. Figure 4a is the orientation map for this sample with the color code shown in Figure 4b. Selected pole figures are also shown in Figure 4c. It is clear that the sample is made up of many randomly oriented grains a few 10's of microns in diameter. This explains the low value of the measured magnetostriction for this sample as very few of the grains have the preferred <100> direction aligned along the axis of the rod. The purpose of the further investigations as described below was to investigate the possibility of inducing preferred texture in the Fe-Ga rods.

3.2. Zone-melted rods

The X-ray diffraction pattern collected from a radial cross section of a 3 mm diameter zone-melted rod is shown in Figure 2b. The pattern collected for this rod is significantly different than the pattern obtained for the as-cast rod. All of the peaks for bcc-Fe are present, but the ratio of peak intensities is not consistent with a randomly oriented sample. The (110) and (211) peaks have nearly disappeared, with the (200) peak dominating the pattern. This suggests that the planes parallel with the sample surface are mostly (200) and that the sample therefore has a preferred <100> orientation along the axis of the rod. The preferential <100> orientation is confirmed by the EBSD data for a radial cross section of the zone-melted sample as shown in Figure 6. The orientation map in Figure 6a shows that the rod is mostly a single large grain with a (100) plane parallel to the surface. There are a few smaller grains around one edge, only one of which does not also have a (100) plane parallel to the surface. The one grain that is significantly different than the others is oriented about 10° from the <100> direction. The (100) pole figure for this sample in Figure 6c also is indicative of a sample with a preferred <100> orientation. The partial rings in the (110) and (111) pole figures indicate that while the sample is predominantly <100> oriented, there is more than one grain and they are rotated to some extent from one another around the axis of the rod (the z-axis for the pole figures).

To check that the grain growth and orientation found for the zone-melted sample was not localized to the cross section shown in Figure 5, EBSD data were collected on a polished axial section. The resulting orientation map is shown in Figure 6, using the same color code as the two previous maps. The section measured was approximately 1 cm in length, and found to be essentially one grain at the polished surface with an orientation of about 10° from <100> (based on the cubic nature of the structure the orientation in an axial cross section would have to be between 0 and 45° from the <100> direction). The combination of the XRD and EBSD data strongly indicates that the zone-melted rod is made up of large grains with the <100> direction aligned very near to the long axis of the rod.

The magnetostriction of the zone-melted rod as measured with the capacitance dilatometer is shown in Figure 7. The zone-melting process increased the magnetostriction by a factor of two (i.e. from 50 to100 ppm) relative to the as-cast rod. The overall shape of the curve is still the same, although there is a slight decrease in the amount of hysteresis present. Some effects due to magnetic history have been seen in the magnetostriction data. These are likely due to energy associated with domain walls (see e.g. [22]) and further investigations of these effects will be reported elsewhere. While the magnetostriction of the zone-melted rod is still somewhat less than the maximum values of around 290 ppm reported for single crystals of similar composition [17], the techniques used to produce and process the rod are much more efficient, less complex and less expensive than the techniques used to obtain single crystals. The measured saturation magnetostriction is expected to increase with Ga content up to about 19%, so the present sample has slightly less than the optimal Ga content. Further improvement may be expected for samples with slightly higher Ga content (~19 at%).

3.3. Zone-melted and annealed rods

The effects of annealing the zone melted rod have also been investigated. Annealing was performed under flowing Ar at a temperature of 650 °C for 4 hours. The magnetization curve for the annealed sample was indistinguishable from that of the as-cast and zone melted rods. The XRD pattern for the annealed rod is shown in Figure 2(c). This is seen to be similar to the XRD of the as-cast rod and clearly shows that the preferred <100> texture introduced by zone melting has been substantially modified by annealing. The details of these changes can be readily seen in the EBSD results for the annealed rod as shown in Figure 8. These results show that the grains are fairly large (although perhaps on the average somewhat smaller than those in the zone melted and unannealed sample). The EBSD results also show that the predominant texture is close to the <111> direction with some component close to the <110> direction. These results readily explain the XRD pattern shown in Figure 2(c). As the <111> peak does not appear for the bcc structure, the remaining most intense peak occurs for the <110> reflection giving rise to an XRD pattern which is similar to the randomly oriented as-cast sample.

Magnetostriction was greatly affected by the annealing as seen in Figure 9. The magnetostriction for the annealed zone-melted rod has decreased from 100 ppm to about 25 ppm. This is consistent with the expectations for a sample with texture along <110> and/or <111> directions [23]. This crystallographic retexturing may be related to surface effects.

4. Conclusions

It has been previously shown that processing techniques can greatly influence the microstructure of Fe-Ga alloys. In particular, rapid quenching from the melt tends to suppress the formation of the $D0_3$ phase, which is believed to be detrimental to the magnetostriction of the alloy. It is believed that for the rods in this work, the suction extraction and zone-melting technique do not allow the formation of the $D0_3$ phase. The production and characterization of <100> oriented Fe-Ga rods has been described. Rods of Fe-Ga can be rapidly produced using a suction extraction technique. Large magnetostriction can be achieved with these rods by a subsequent directional solidification by the zone-melting process using translation rates that are substantially greater than those reported previously in the literature [17]. Polycrystalline rods, 3 mm in diameter, with a preferred <100> orientation along the rod axis and a maximum magnetostriction of 100 ppm have been produced by this method. Annealing zone-melted rods resulted in a reduction of the saturation magnetostriction due to a reorientation of the crystallographic texture. The method described in the present paper provides a straightforward and cost effective method of producing crystallographically oriented bulk Fe-Ga materials with a preferential <100> texture giving rise to an enhancement of the magnetostrictive properties. This provides a basis for further studies of the effects of thermal treatment parameters and alloy composition in order to maximize the usefulness of the resulting materials.

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Figure Captions

Figure 1. Photograph of two Fe-Ga rods produced by suction extraction.

Figure 2. XRD pattern of radial cross sections of (a) as-cast rod [data not collected above 2θ = 65°], (b) zone-melted rod and (c) zone melted and annealed rod.

Figure3. Magnetostriction, in parts per million, versus applied field for the as-cast Fe-Ga rod in Figures 2 and 3.

Figure 4. EBSD data for a radial cross section of the as-cast rod showing (a) orientation map, (b) color code and (c) selected pole figures. The scale bar in (a) is 700 μm.

Figure 5. EBSD data for a radial cross section of the zone melted rod showing (a) orientation map, (b) color code and (c) selected pole figures. The scale bar in (a) is 700 μm. Note: The oval image of the circular rod results from the fact that the incident electron beam is not normal to the surface of the sample.

Figure 6. Orientation map for an axial cross section of the zone melted rod of Figures 2 and 3 using the same color code and grain boundary markers as Figure 5. The scale bar is 3000 μm.

Figure 7. Magnetostriction, in parts per million, versus applied field for the zone-melted Fe-Ga rod.

Figure 8: EBSD orientation map (top) and pole plots (bottom) for a radial cross section of a rod that has been zone melted and subsequently annealed. The color code and grain boundary markers are the same as in Figure 5.

Figure 9. Magnetostriction versus applied field for a zone-melted and annealed Fe-Ga rod.

Figure 1.

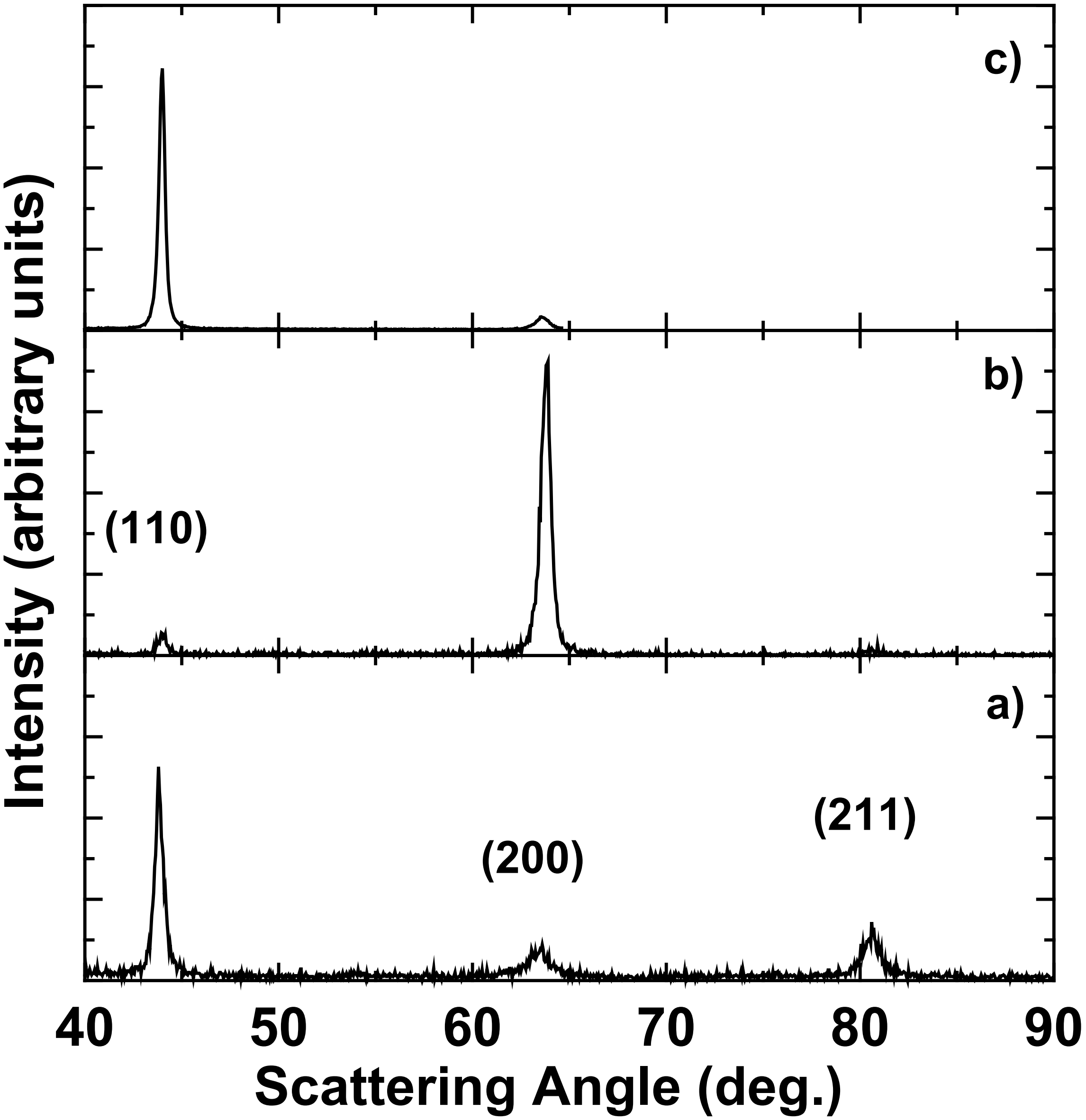
c)
b)
a)
(110)
(200)
(211)
Intensity (arbitrary units)
40
50
60
70
80
90
Scattering Angle (deg.)

Figure 2.

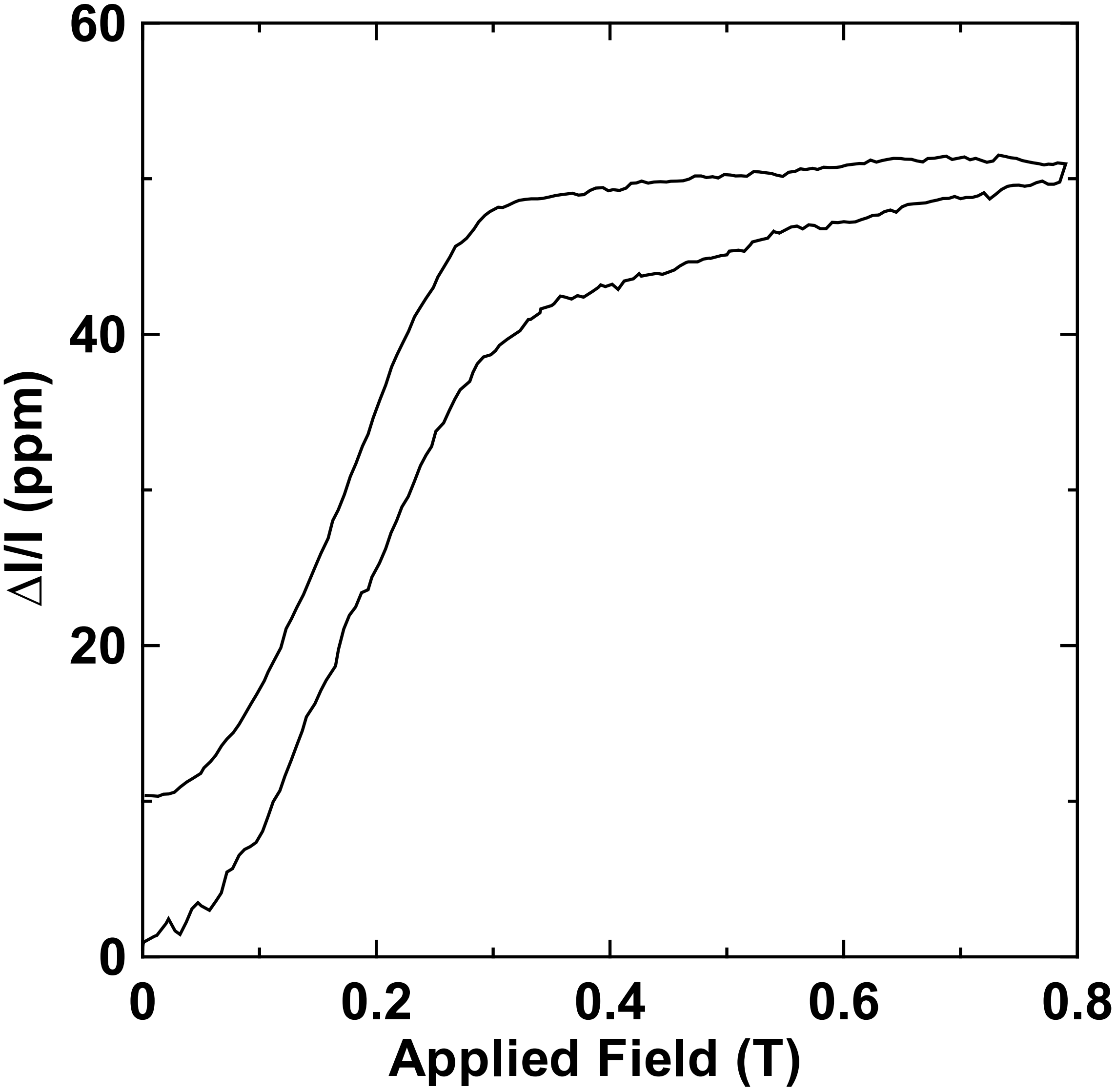

Figure 3.

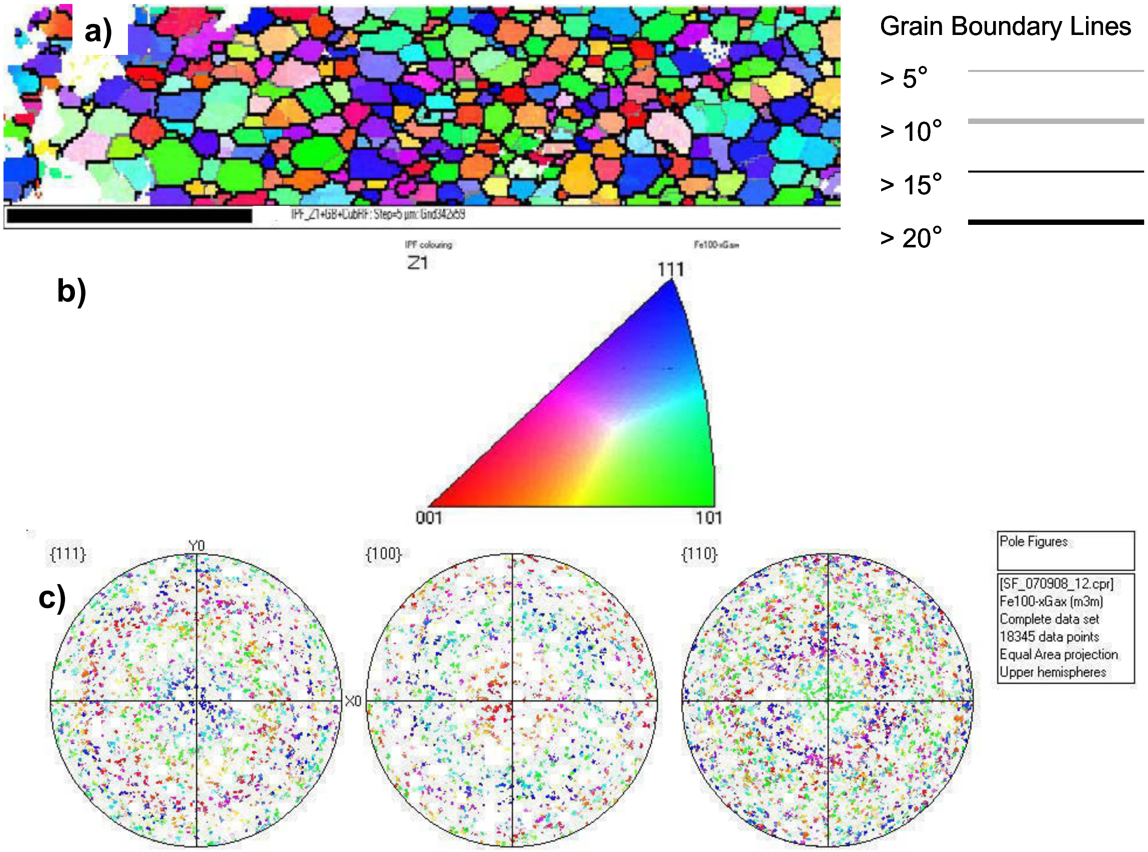
a)
IPF_Z1+GB+CubRF: Step=5 µm: Grid342x69
Grain Boundary Lines
> 5°
> 10°
> 15°
> 20°
b)
IPF colouring
Z1
Fe100-xGax
111
001
101
c)
{111}
Y0
X0
{100}
{110}
Pole Figures
[SF_070908_12.cpr]
Fe100-xGax (m3m)
Complete data set
18345 data points
Equal Area projection
Upper hemispheres

Figure 4.

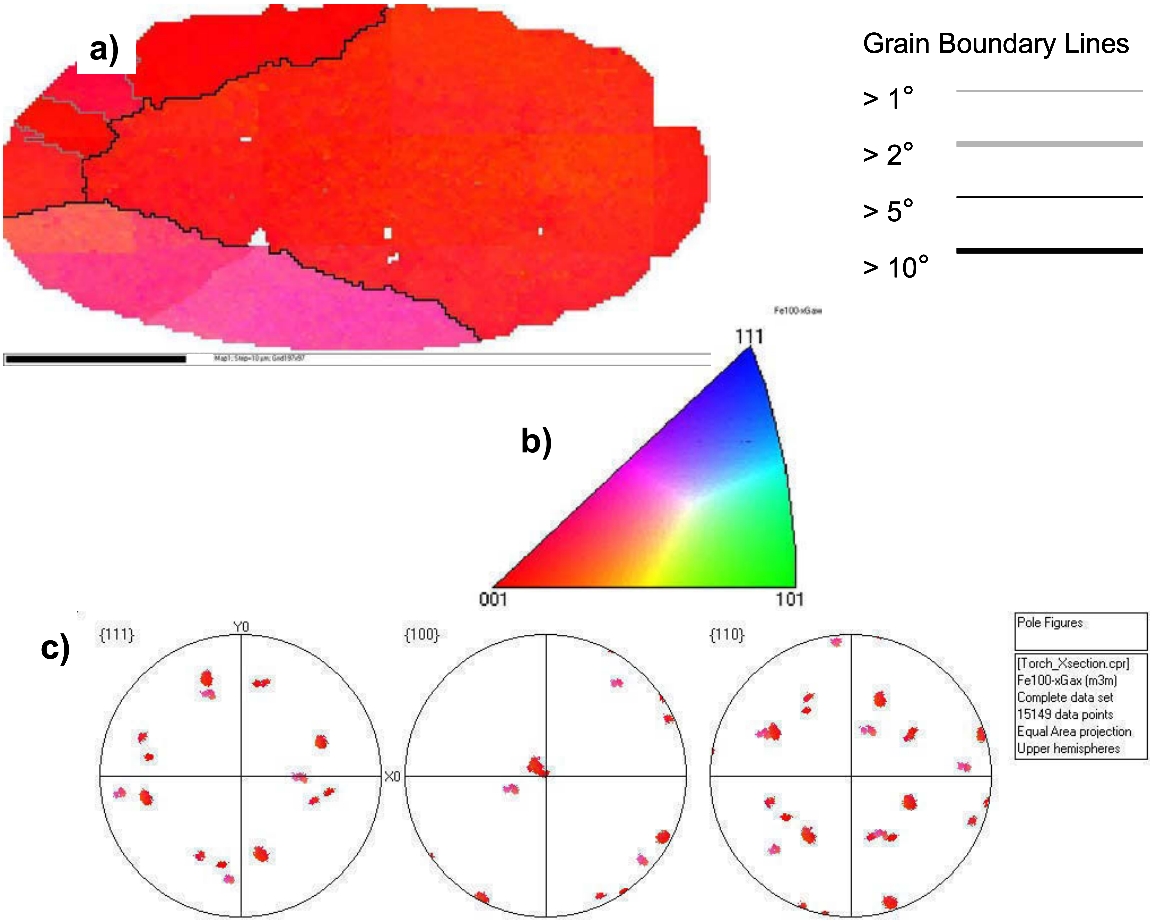
a)
Grain Boundary Lines
> 1°
> 2°
> 5°
> 10°
Map1: Step=10 µm; Grid197x97
Fe100-xGax
b)
111
001
101
c)
{111}
Y0
X0
{100}
{110}
Pole Figures
[Torch_Xsection.cpr]
Fe100-xGax (m3m)
Complete data set
15149 data points
Equal Area projection
Upper hemispheres

Figure 5.

Figure 6.

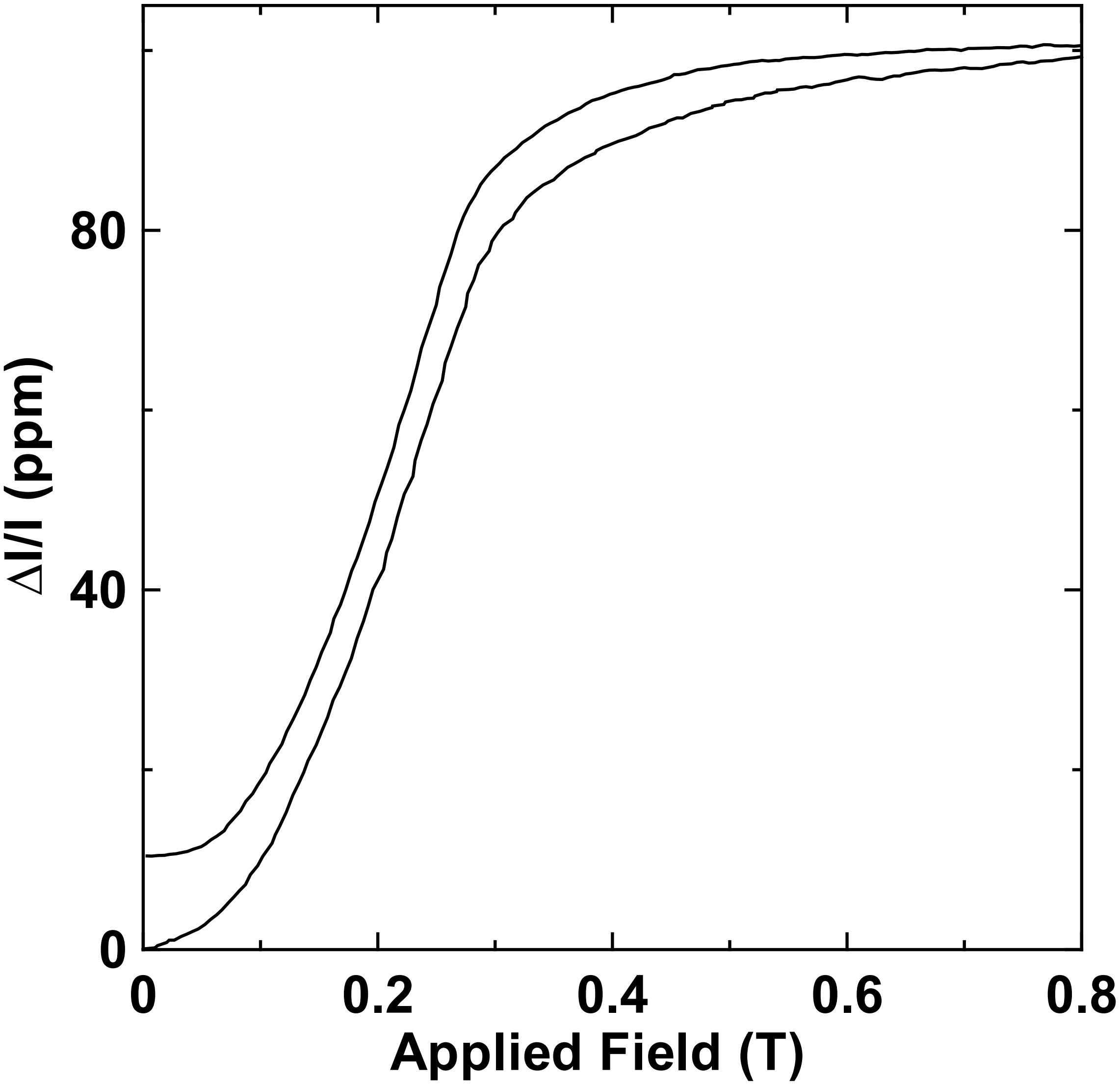

Figure 7.

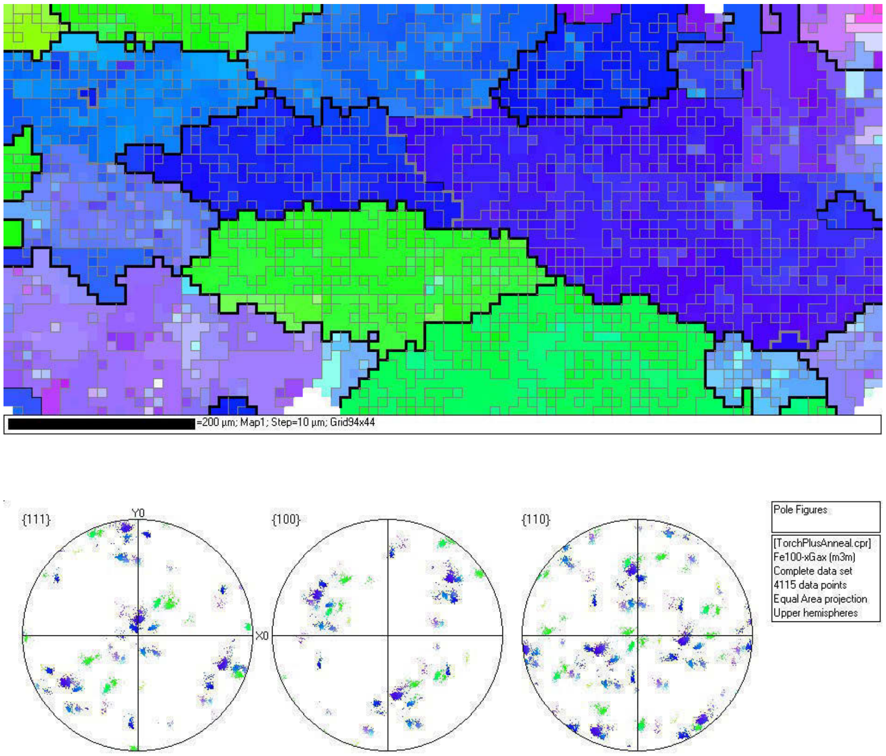
=200 µm; Map1; Step=10 µm; Grid94x44
{111}
Y0
{100}
X0
{110}
Pole Figures
[TorchPlusAnneal.cpr]
Fe100-xGax (m3m)
Complete data set
4115 data points
Equal Area projection
Upper hemispheres

Figure 8.

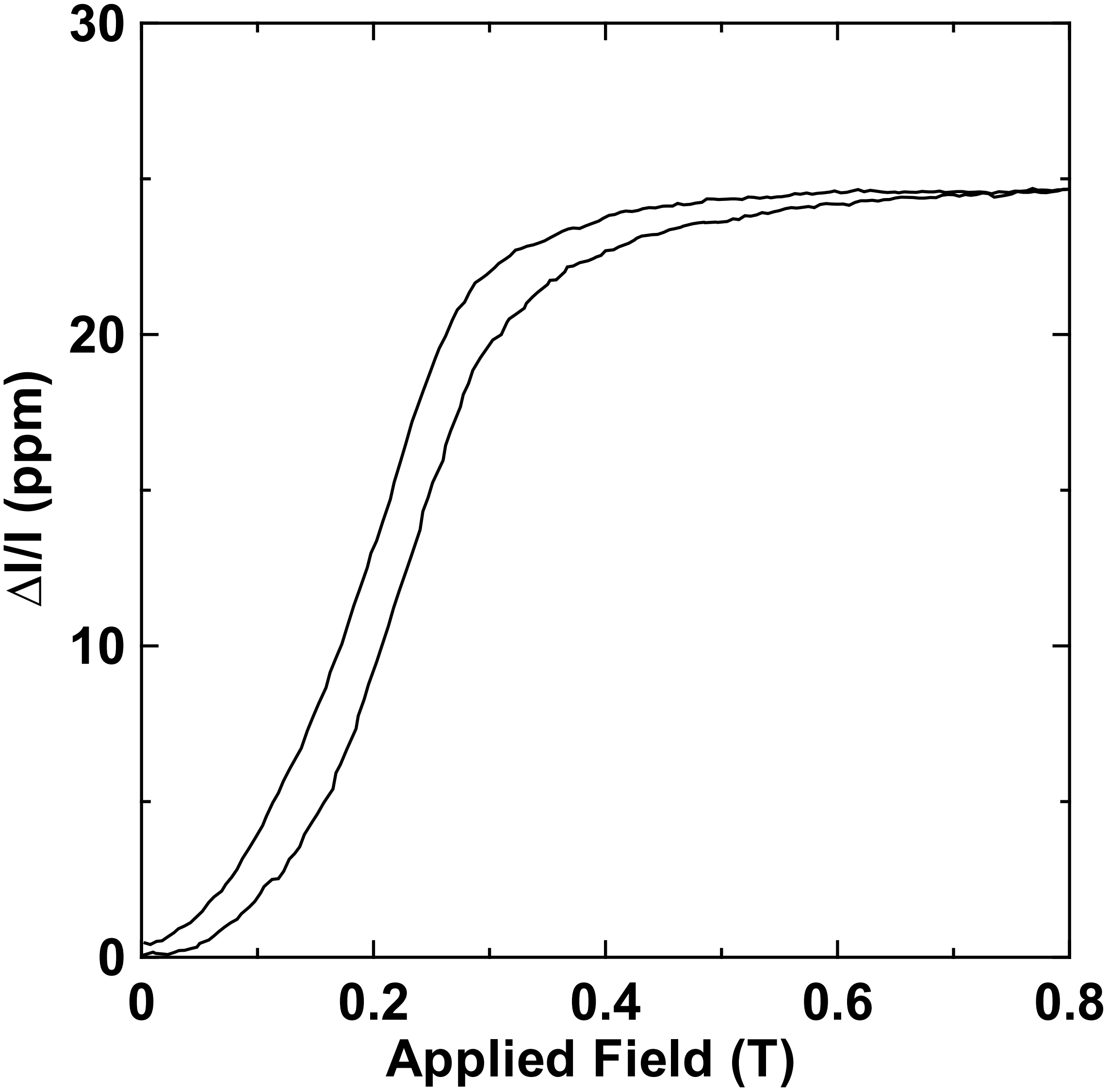

Figure 9.

Annex B Publications and Presentations

Journal papers related to this research

1. T.D. Hatchard, A.E. George, S.P. Farrell, M.O. Steinitz, C.P. Adams, M. Cormier and R.A. Dunlap, *Production and characterization of <100> textured magnetostrictive Fe-Ga rods.* Journal of Alloys and Compounds, 494; (2010), p. 420-425. (DRDC Atlantic SL 2009-144; May 2009).
2. S.P. Farrell, P.E. Quigley, K.J. Avery, T.D. Hatchard, S.E. Flynn and R.A. Dunlap, *Development of <100> crystallographic texture in magnetostrictive Fe-Ga wires using a modified Taylor wire method.* Journal of Physics D: Applied Physics, 42 (2009) p. 135005. (DRDC Atlantic SL 2008-227; October 2008).
3. T.D. Hatchard, J.S. Thorne, S.P. Farrell and R.A. Dunlap, *Production of Ni100-x-yMnxGay Magnetic Shape Memory Alloys by Mechanical Alloying.* Journal of Physics: Condensed Matter, 20 p 445205-12, 2008. (DRDC Atlantic SL 2008-047,).
4. J.M. Gaudet, T.D. Hatchard, S.P. Farrell and R.A. Dunlap, *Properties of Fe-Ga based powders prepared by mechanical alloying*. Journal of Magnetism and Magnetic Materials, 320, p 821-829, 2008. (DRDC Atlantic SL 2007-143, June 2007,).
5. S.P. Farrell, P.E. Quigley, K.J. Avery, T.D. Hatchard, S.E. Flynn and R.A. Dunlap, *Magnetostrictive Fe-Ga Wires with <100> Fiber Texture*. Materials Research Society Symposium Proceedings, 1129, 6 pages 2009. (DRDC Atlantic SL 2008-225; October 2008)
6. J.M. Gaudet, T.D. Hatchard, S.P. Farrell and R.A. Dunlap, *Magnetostrictive Fe-Ga alloys prepared by mechanical alloying*. Proceedings of the “2007 CF/DRDC International Defence Applications of Materials Meeting”, June 5-7, 2007, Halifax NS, 10 pages (DRDC Atlantic SL 2007-165, September 2007)
7. T.D. Hatchard, J.S. Thorne, S.P. Farrell and R.A. Dunlap, *Production of* $Ni_{1-x-y}Mn_xGa_y$ *Magnetic Shape Memory Alloys by Mechanical Alloying.* Proceedings of the “2007 CF/DRDC International Defence Applications of Materials Meeting”, June 5-7, 2007, Halifax NS, 11 pages (DRDC Atlantic SL 2007-166).
8. J.S. Thorne, S.P. Farrell, T.D. Hatchard and R.A. Dunlap, *Short Range Structural Order in Melt Spun Fe-Ga Alloys*. Proceedings of CanSmart 2006- 9th Cansmart Meeting International Workshop on Smart Materials and Structures, 12-13 October 2006, Toronto, Ontario. (DRDC Atlantic SL 2006-191, September 2006).

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Magnetostrictive alloys are novel ‘smart’ materials that exhibit dynamic coupling of magnetic and mechanical properties. The dynamic magnetic-mechanical coupling phenomenon may make magnetostrictive alloys useful for transforming kinetic energy into magnetic energy to be harvested for electrical energy (and vice versa). This offers the potential for magnetostrictive alloys to be useful for development of efficient, cost-effective transducer, actuator, smart structure, vibration dampening and energy harvesting materials for military applications.

Several low-cost processing approaches that produce highly-textured thin-form magnetostrictive Fe-Ga alloys were found to be promising for energy harvesting applications. Dalhousie University was contracted to evaluate several techniques for the production of magnetically active alloys and to evaluate the resultant thermal, magnetic and crystallographic properties of the alloys. This document summarizes the principal results from the final two years of the project and focuses primarily on development of Fe-Ga magnetostrictive alloys. Mechanical alloying (via ball milling) and rapid quenching (ribbon casting) were shown to be cost-effective methods for producing Fe-Ga compositions. The magnetic and crystallographic structures were fully characterized and show unique properties. ^{57}Fe Mössbauer hyperfine fields were shown to be particularly sensitive to atomic scale clustering (as a precursor to ordering). Highly-oriented feedstock Fe-Ga rods have been produced using the cost effective suction extraction method. Fe-Ga rods provided excellent feedstock material that improved the quality, ie., <100> texture, of DRDC’s fabricated Fe-Ga magnetostrictive wire.

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Energy Harvesting, Magnetostrictive alloys, Fe-Ga alloys

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