Journal Pre-proof

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PII: S0925-8388(21)00145-6

DOI: https://doi.org/10.1016/j.jallcom.2021.158738

Reference: JALCOM158738

To appear in: *Journal of Alloys and Compounds*

Received date: 11 September 2020
Revised date: 8 January 2021
Accepted date: 10 January 2021

Twinning-induced large tensile strains in directionally-solidified $Fe_{100-x}Ga_x$ oligocrystals

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$Fe_{100-x}Ga_x$ alloys require highly textured samples for optimal magnetostrictive performance, which is often accomplished through directional solidification. In this study, we measure the mechanical properties of commercial $Fe_{81.6}Ga_{18.4}$ oligocrystals using sub-sized tensile specimens and digital image correlation (DIC). While many samples fractured around 360 MPa (the literature yield/tensile stress), most had discontinuous yielding as low as 186 MPa. Many of the samples also exhibited surprisingly large tensile strains, up to 5%, with substantial specimen-to-specimen variability. In all specimens, DIC showed high strain bands that develop at very defined angles, in agreement with electron back-scattered diffraction, which revealed significant deformation twinning.

***Keywords* — Twinning; Digital Image Correlation (DIC); tension test; Iron Alloys; Magnetostriction**

1. Introduction

Magnetostrictive alloys are materials which exhibit strain when in the presence of a changing applied magnetic field. While there are many applications for these alloys, most compositions require operation under compressive stresses. This is because magnetostrictive materials are generally brittle and have a low fracture strength in tension, similar to most piezoelectric materials. $Fe_{100-x}Ga_x$ (Galfenol) alloys, however, can operate under tensile loads while still exhibiting large magnetostrictive strain, with an ultimate tensile strength of ~360 MPa (52 ksi) [1] and with no loss in magnetostrictive performance up to ~30 MPa (4.4 ksi) after compression annealing [2]. This tensile operability opens up many new avenues for the design of magnetostrictive devices, especially in applications where buckling is of concern due to material geometry.

This improved mechanical performance comes with a reduction in magnetostrictive performance compared to heavy rare earth alloys, such as Terfenol-D, and so device manufacturers have sought to maximize the magnetostriction of their Galfenol products to overcome this loss in achievable strain. Magnetostriction would be maximized in a single-crystal component with the crystal orientation strategically aligned along the direction of actuation, but true single crystals are impractical to produce. Instead, manufacturers often produce components comprised of a few very large grains (~5 mm in diameter) that are closely aligned in order to approach single-crystal performance. Due to the inclusion of gallium, these samples suffer from liquid metal embrittlement [3] and weak grain boundary cohesion strength [4]. The weakened mechanical properties, coupled with the very low tortuosity of the grain boundaries in these directionally-solidified oligocrystalline components, results in a material which can fail catastrophically via intergranular fracture with no plastic deformation [5], without proper microstructure engineering.

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This is obviously not desirable for applications under tension, especially in applications where variable loads may occur.

In the present work, we have evaluated the tensile properties of commercial oligocrystalline Galfenol using digital image correlation (DIC). DIC is a strain measurement technique that uses a succession of digital images to obtain incremental strain fields of a planar surface [6]. It is a non-contact method, gives full field strain measurements, and can be used on a range of length scales [7]. It also offers unique advantages for measuring strain in low-ductility oligocrystalline materials when compared with a load frame's linear variable differential transformer (which can be a misleading measure of specimen gage length strain), extensometers (which can result in the material fracturing prematurely near the extensometer knife edges) and strain gauges (which can separate from the specimen due to moderate strains or grain pop-off).

2. EXPERIMENTAL PROCEDURE

2.1. Material Fabrication

Directionally solidified rods of Fe–18.4 at% Ga were supplied by ETREMA Products, Inc (permanently closed; material production taken over by TdVib, LLC). These rods were fabricated by the free-stand zone melting (FSZM) technique, and were subsequently compression annealed. They were then machined, using electrical discharge machining (EDM), into sub-size tensile specimens 101.6 mm (4 in) long and 3.0 mm (0.12 in) thick, with a gage section width of 6.4 mm (0.25 in) and a gage length of 25.4 mm (1 in), in accordance with ASTM Standard E8/E8M [8]. All evaluated samples were found free of internal and external defects through visual inspection, dye penetrant testing, and radiographic testing.

2.2. Mechanical Testing

Seven samples were pulled to fracture in a 100 kN (22 kip) MTS Systems Corporation servo-hydraulic load frame at a quasi-static displacement rate of 0.00254 mm/s (0.0001 in/s). The strain for each sample was measured using DIC. The DIC method utilized two stereoscopic cameras and a speckled pattern on the surface of the sample (Fig. 1), which was calibrated using an array of dots with known spacing. To prepare the speckle pattern, the test area surface was first cleaned, and a single coat of white flat spray paint was applied to the surface. A random speckle pattern was then applied using black flat spray paint. Images were recorded using Correlated Solutions Vic Snap software program [9] at a rate of one image/second. The DIC images were post-processed using Correlated Solutions Vic 3D system [9] to extract the full field strain based on the images taken during testing; tensile strain was calculated by averaging over a centralized rectangle. Audible responses from the specimen were also noted during testing, to correlate with any artefacts in the measured stress-strain curves.

2.3. Post Mortem Microstructure Characterization

Fractured DIC samples were cut and mounted in a Struers bakelite hot mounting resin with carbon filler, ground and polished by traditional metallurgical techniques, and finished with vibratory polishing in a 0.04 μm colloidal silica suspension. The DIC speckled surface was used as the imaging surface for crystallographic orientation. Electron back-scattered diffraction (EBSD) was performed in a Hitachi SU6600 scanning electron microscope (SEM) equipped with EDAX OIM 7 software to create inverse pole figures for crystallographic orientation determination.

3. RESULTS AND DISCUSSION

While not all specimens achieved the literature ultimate tensile strength (360 MPa, 52 ksi), the average fracture stress of the seven samples tested with DIC was 385 MPa (56 ksi), with a maximum of 435 MPa (63 ksi) and a minimum of 313 MPa (45 ksi). Some of these specimens also exhibited total elongation up to 4.8% (with a sample average of 2.1%). The specimens, however, exhibited discontinuous yielding and serrations in their stress-strain curves, which are exemplified in Fig. 2. While these serrations usually occurred at a stress which was approximately equal to the published yield strength for this material, some serrations occurred at a stress as low as 186 MPa (27 ksi), and continued to occur at increasingly higher stresses until fracture. Each one of these drops in stress coincided with an audible response, as also observed by Yusada et al. for a single crystal, Fe–23.8 at. % Ga alloy [10]. Individual grains would occasionally pop off of the specimens, as shown on the right side of Fig. 1.

The DIC full field strain contours provide additional insights into this mechanical behavior. As the applied stress increases, initially the specimen strains fairly uniformly. In all specimens, at various critical stress values, however, bands of increased local strain appear; Fig. 3 shows a set of DIC images from a representative sample up to fracture, with the time stamp and applied stress value indicated. The critical stresses, band width and longevity depend upon the crystal orientation of the sample. As the stress increases, more bands appear, all of which are inclined at ~60° from the tensile axis (see the Supplementary material for two example videos). The appearance of these bands coincides with the audible events mentioned above, and bands remain visible until fracture. Interestingly, the fracture often occurs outside of the high strain bands, even though those bands are the apparent locations of strain concentration. Initially, one may ascribe these strain bands to Lüders bands [11], however, Lüders bands are associated with waves of plastic strain moving through the material, and do not apply in this case.

Further analysis of these strain bands was performed using EBSD to probe the crystallographic orientations of the specimens. Fig. 4 shows an EBSD inverse pole figure (IPF) map montage of one of the specimens showing strain bands. Interspersed throughout the gage length (which in this region of the specimen consists of a single parent grain), there are also a large number of deformation twins. Due to the very large grains in these specimens, many of the twins were able to traverse the entire width of the tensile specimen, which would be accompanied by a substantial load drop upon forming; since the stress-strain curve was measured under displacement control, the formation and growth of the deformation twin slightly elongates the sample, reducing the amount of stress needed to maintain the desired sample elongation. The audible response from the specimens during testing can also be attributed to this coarse twinning, which is akin to the well-known "tin cry" phenomenon and which has been observed in similar alloys [10, 12]. It is interesting to note that in some cases multiple deformation twin variants were formed within a grain, while in other cases only a single variant could be observed across very large areas of the specimen. All observed variants had a ~60° misorientation with its parent grain, however, which is consistent with {112}<111> twinning observed in similar alloys [10, 12]; if one rotates the EBSD IPF (Fig. 4) by 36°, the deformation twins line up well with the DIC strain banding (Fig. 3), at both +55° and -66° inclinations from the loading axis. Large variations in twin thickness were also observed between specimens, ranging from <1 μm to nearly 30 μm, although the latter may have originated as several adjacent twins which grew and consolidated during deformation.

In twinning-induced plasticity (TWIP) steels, twins are known to play a very important role in improving uniform ductility, although this improvement comes about mainly because of the twin's impact on dislocation density in the surrounding material and not because of the strain introduced by the twin itself [13]. It is likely that the twins formed in this alloy system play an important role in minimizing stress concentration and localized failure also, although the interactions between twins and dislocations have not been evaluated here. In this context, it is not surprising that the specimens typically fractured in areas where the local strain was comparatively small (i.e., where the specimen was unable to relieve stress concentration by twinning). The deformation twins provide a mechanism of plastic deformation in an otherwise brittle alloy; once these opportunities for strain are exhausted, the sample fractures catastrophically. Controlling the stacking fault energy of the material through alloying additions could further increase the propensity for twinning and thereby improve ductility, as was done using Al in Ref. [12].

While most current magnetostrictive applications would not approach the yield stress of this material, it is important to understand the unique strain behavior and the lack of uniformity in performance below the ultimate tensile stress due to unexpected loading in harsh environments. Future work should focus on increasing the strengthening and toughening mechanisms available in this alloy system without sacrificing the component's achievable magnetostrictive strain, in order to increase the design window for these active structural materials into the realm of structural applications. Such improvements might be achieved through a reduction in the grain size, which functionally shortens the length of twins, and an increase of grain boundary cohesion strength. Coupled with a greater understanding of active deformation mechanisms, these might eventually provide a more consistent and higher yield strength with greater ductility (>10%), with a switch in fracture mode from intergranular to transgranular fracture.

4. CONCLUSIONS

Fe–18.4 at% Ga oligocrystalline specimens were fractured under tensile stress, with strains being measured by DIC. The full field strain contours of DIC showed strain bands at specific angles, which can be attributed to the development of deformation twins as seen through EBSD. These deformation twins allowed the material to accommodate moderate amounts of plastic strain, up to ~5%. By understanding the deformation mechanisms of Fe-Ga alloys, in addition to the origins of large magnetostriction, it becomes possible to transition these transduction alloys to more structural applications, while maintaining their multifunctional properties. Rather than using magnetostrictive alloys as a sensing component added to a piece of the structure, it becomes plausible to have the magnetostrictive alloy act as the structure itself. Similar future studies on polycrystalline rolled sheets of Fe–18.4 at% Ga will be performed to understand the roll of twinning in smaller grained samples, and to observe any potential smoothing of the stress-strain curve due to deformation twins which are smaller in length, inhibited by the presence of additional grain boundaries.

Declaration of Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This work was supported by the U.S. Office of Naval Research (Code No. 321MS, Maritime Sensing, Grant No. N0001416WX00164). We would like to thank Eric Summers of ETREMA Products, Inc., Ames, IA for supplying the samples. We would also like to thank Ashlee Floyd and Joel Benton of the Welding, Processing and Non-destructive Evaluation Branch, Naval Surface Warfare Center, Carderock Division for their help in dye penetrant testing and radiographic testing, respectively, and our Science and Engineering Apprenticeship Program (SEAP) intern, Sydney Wolk, for her assistance in the dye penetrant and mechanical testing.

Disclaimer

Uncertainty quantification was outside of the scope of this paper.

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CAPTIONS

Fig. 1. Fractured Galfenol tensile specimen, showing an example speckle pattern, and a shiny, brittle fracture surface.

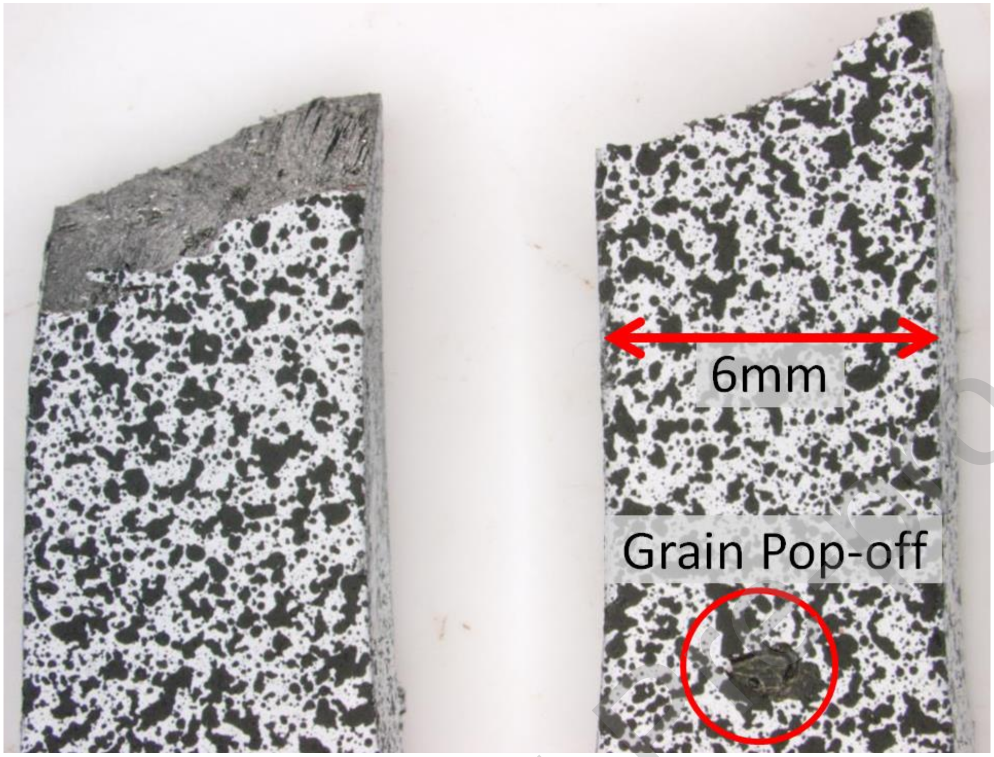


Fig. 2. Tensile stress in an Fe–18.4 at% Ga sub-size specimen plotted against the strain of the sample as measured through digital image correlation (DIC).

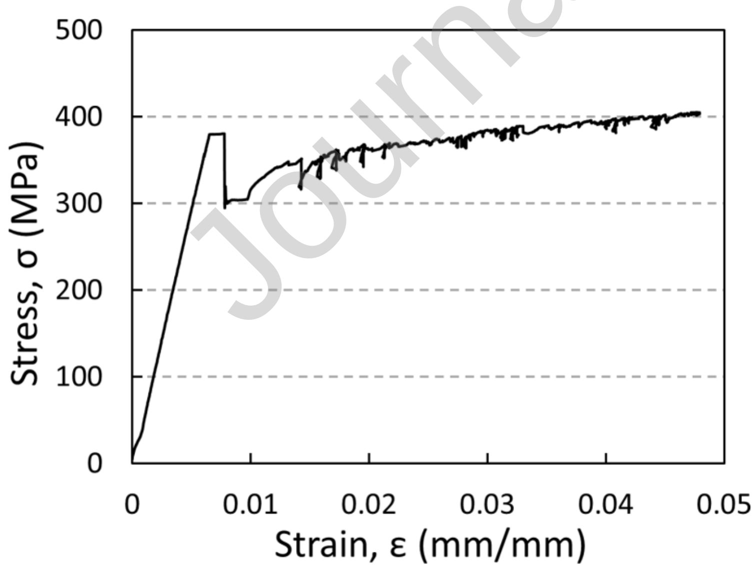


Fig. 3. A set of full field strain contour maps of an $Fe_{81.6}Ga_{18.4}$ sub-size dog-bone specimen pulled under tension, as measured through Digital Image Correlation (DIC), with the time stamp and applied stress value during the test indicated below each image. Strain bands can be seen at very specific angles (+56° and -66° from the tensile axis), which can also be attributed to the drops in stress and audible pings during testing.

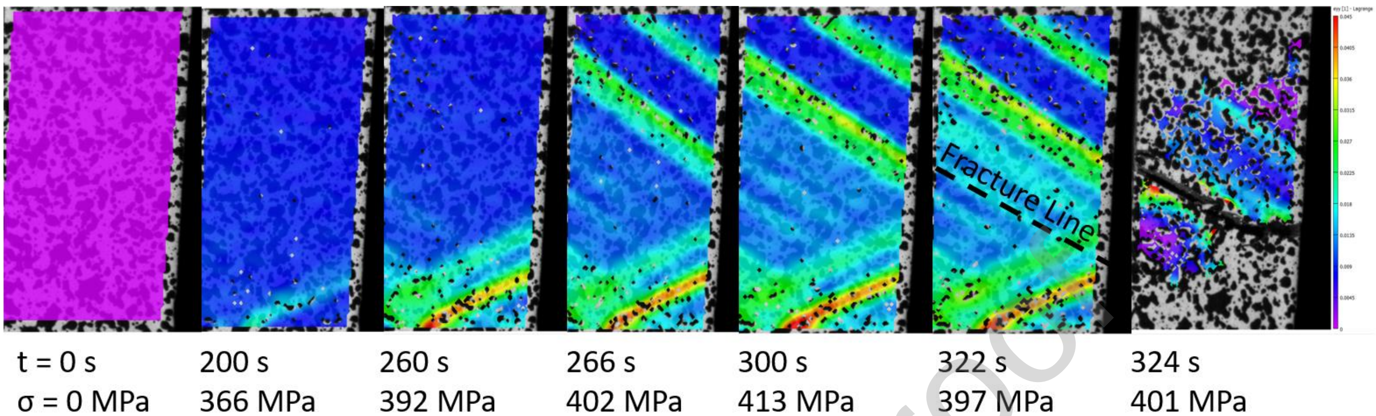
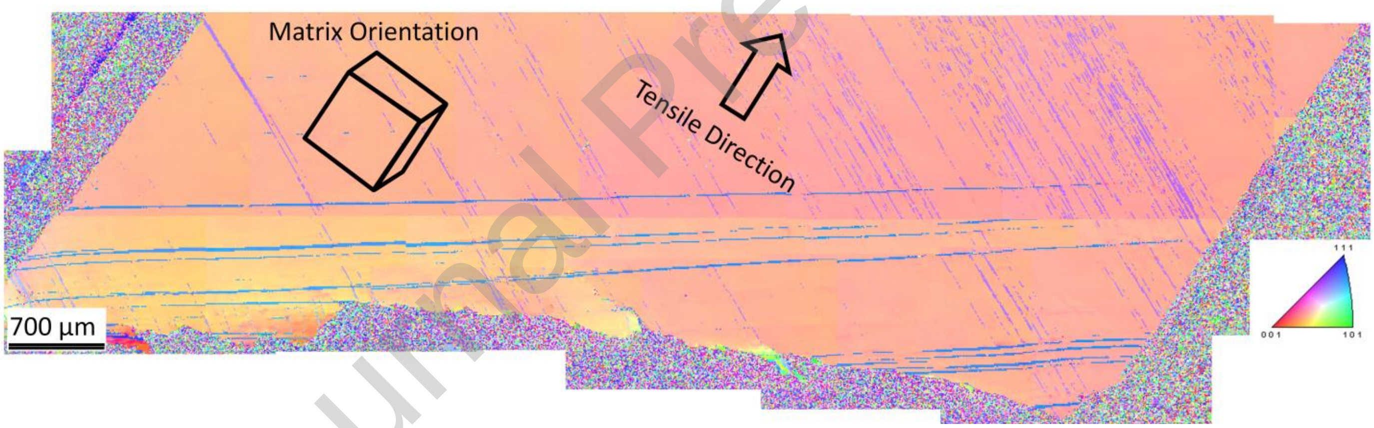
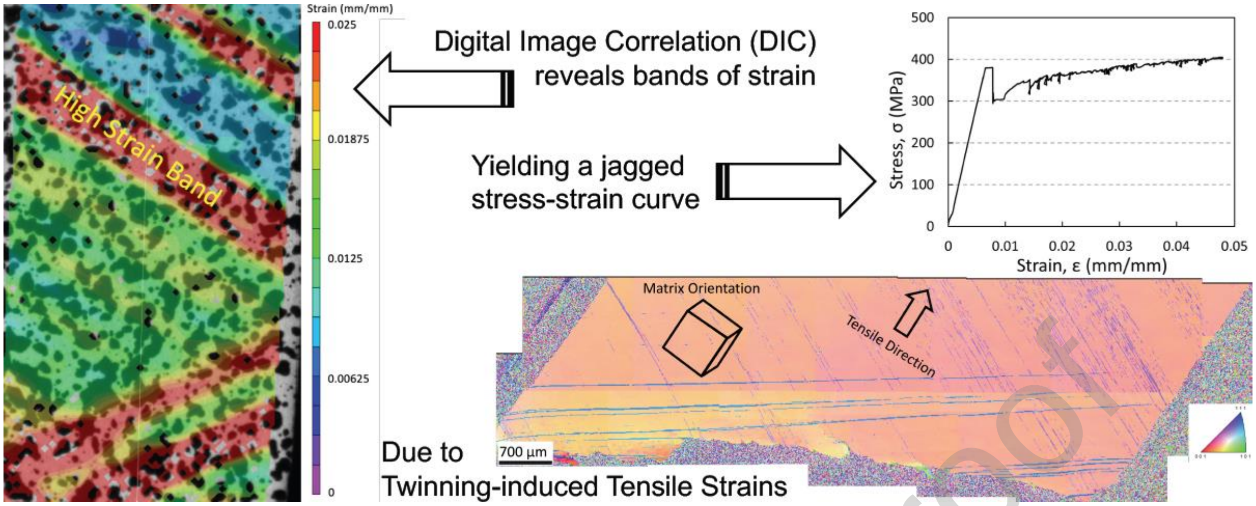


Fig. 4. Inverse pole figure map montage of the fracture surface of an $Fe_{81.6}Ga_{18.4}$ sub-size dog-bone specimen as measured through electron backscatter diffraction (EBSD). The matrix has one crystallographic orientation between the (100) and (110) planes (as illustrated), with many deformation twins traversing the entire width of the specimen.



Graphical abstract



Credit Author Statement

Nicholas J. Jones: Conceptualization; Data curation; Formal analysis; Funding acquisition; Methodology; Roles/Writing – original draft; Writing - review & editing

Paul K. Lambert: Formal analysis; Methodology; Visualization; Roles/Writing – original draft; Writing - review & editing

Yared Amanuel: Conceptualization; Data curation; Formal analysis; Methodology; Visualization; Roles/Writing – original draft

azalyn D. Dukes: Data curation; Formal analysis; Visualization; Roles/Writing – original draft **Jin-Hyeong Yoo:** Data curation; Formal analysis; Roles/Writing – original draft

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Highlights

- Large-grained FeGa alloys exhibited discontinuous yielding
- Despite catastrophic failure, alloy exhibited strains up to 5%
- Strain banding observed during mechanical testing using Digital Image Correlation
- Observed strain is due to twinning induced plasticity