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Simultaneously improving mechanical and magnetic properties through heterogeneous lamella structures in a superalloy-like, soft magnetic complex concentrated alloy

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ABSTRACT

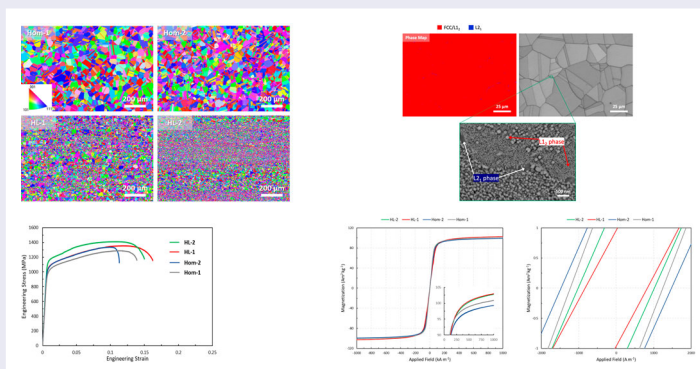
Heterogeneous lamella (HL) structures were produced via simple processing steps in a FeCoNi-based complex concentrated alloy (CCA) with superalloy-like FCC/L1₂ microstructure. Compared to the homogeneous-structured control specimens, the HL-structured specimens exhibited synergistic enhancements in strengths and ductility (with σ_y up to 1 GPa, σ_u up to 1.4 GPa, and ϵ_t up to 16.5%), and at the same time, higher saturation magnetization and lower coercivity (with M_s up to 104 Am² kg⁻¹ and H_c as low as 833 A/m). The results obtained in this study provide insights to enhancing mechanical properties in structural soft magnetic materials without sacrificing their magnetic properties.

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KEYWORDS

Complex concentrated alloys; Coherent precipitation; Heterogeneous lamella structure; Mechanical properties; Magnetic properties



IMPACT STATEMENT

The current work presents a simple and effective strategy that can be applied to soft magnetic alloys with superalloy-like microstructures for simultaneous enhancements of mechanical and magnetic properties.

Introduction

The strategy of alloy design has experienced a paradigm shift since the emergence of high-entropy alloys (HEAs) in 2004. Until then, the same alloy design concept that had been utilized ever since the bronze age, had still been employed to develop modern alloys [1]. Due to the compositional and property limitations of this approach (specific trace amounts of alloying elements are added to less than two principal elements), the performance improvement of traditional alloys had reached a bottleneck [2]. The discovery of HEAs freed alloy compositions

from the corners of phase diagrams into the vast central region and brought numerous possibilities for new alloys with novel properties. Over the past two decades, HEAs have evolved and expanded to complex concentrated alloys (CCAs) or multi-principal element alloys (MPEAs), which possess even more compositional and microstructural complexities to better meet the needs of new alloys for extreme service conditions [3].

One of the typical microstructural types of CCAs that has received much research attention is the superalloy-like FCC/L1₂ structure [4–22] due to their resemblance

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to the traditional Ni-based superalloys which are known for their high strength and ductility, at both room and elevated temperatures. Combining the extra strengthening effects intrinsic to CCAs [23] and the intriguing properties found in the multicomponent L_{12} nanoprecipitates [13,15], as well as the numerous possibilities in compositional design, the superalloy-like CCAs have exhibited promising potential for becoming next generation structural materials. On the other hand, CCAs possessing such microstructure usually contain high concentrations of ferromagnetic elements such as Fe, Co, and Ni [4–9,11–13,15–22], which can endow them with the potential for possessing good soft magnetic properties [9,11,24–27]. CCAs with both good mechanical and magnetic properties are highly desirable candidates for electric systems operating under high loads such as electric motors and electromagnets [9,25,27].

In recent years, several studies have overcome the well-known strength-ductility trade-off [3,28,29] in strengthening the superalloy-like CCAs, including composition tuning [10,12,15,20], microstructure optimization [6,8,19,21], and utilization of heterogeneous lamella (HL) structure [7,14]. An HL structure is the most effective type of heterostructure in promoting strength-ductility synergy in both traditional metallic materials and CCAs [30,31] and have been successfully applied to multiple CCA systems [7,14,32–38]. The high efficacy and low compositional dependency make HL structures worth further exploration in the current system. The present study has produced HL structures in a FeCoNi-based superalloy-like CCA and first examined their effects on the magnetic properties.

Materials and methods

Alloy ingots with composition of $Al_5Ti_7(FeCoNi)_{86}Cu_2$ (at. %) were produced by arc-melting of raw elements with purities over 99.9% (wt. %) under a Ti-gettered argon atmosphere. Each ingot was flipped and remelted at least 5 times to ensure chemical homogeneity. The ingots were cut into approximately $23 \times 6 \times 10.5$ mm³ rectangular prisms by a diamond saw and homogenized at 1200°C for 2 h followed by quenching into ice water. Each homogenized section was cold rolled (parallel to the 23 mm direction) with a reduction in thickness (RIT) of $\sim 90\%$ into ~ 1 mm-thick sheets. The rolled sheets were then sectioned into ~ 45 mm-long segments by diamond saw and annealed at different conditions for recrystallization followed by water-quenching. The recrystallized samples were aged at 825°C for 3.5 h to allow the precipitation of coherent L_{12} phase. Tensile specimens with gauge length of 18 mm and cross-section of 1.8×1 mm² were waterjet-cut out of the heat-treated sheets

and carefully ground and polished to a mirror surface. Room temperature tensile tests were performed at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ with strain gauges attached on the specimens. At least two specimens for each condition were tested to ensure the reproducibility of the results. Additional loading-unloading-reloading (LUR) cyclic tensile tests were performed at the same strain rate for measuring the hetero-deformation-induced (HDI) stress. The magnetic hysteresis loops were measured at room temperature using a Quantum Design vibrating sample magnetometer (VSM). The measured curves were corrected through post-processing to reflect the magnetization behavior more accurately. Bulk specimens with a dimension of approximately $5 \times 5 \times 1$ mm³ were used for the measurements.

Phase identification via X-ray diffraction (XRD) was conducted on a Rigaku Miniflex II X-ray diffractometer with Cu K α radiation. Microstructure characterizations were carried out using a Thermo Scientific Apreo scanning electron microscope (SEM) equipped with an Oxford Symmetry CMOS electron backscatter diffraction (EBSD) detector. The average grain size and geometrically necessary dislocation (GND) density map were generated through post-processing the EBSD data using Oxford Instrument's Aztec and Tango software.

Results and discussion

Figure 1a visualizes the thermo-mechanical processing route described earlier for achieving homogeneous and HL grain structures with L_{12} nanoprecipitation. The mechanism for forming HL structures in this type of CCA has been explained in-detail in Zhang et al.'s previous work [35]. The XRD plot in Figure 1b shows identification of the main phases, FCC and L_{12} , in the material. The small peak marked by the solid square around 45 degrees matches a minor grain boundary L_{21} phase which has been reported by Yang et al. in similar compositional spaces [15,19]. More detailed phase identification information is provided in the Appedices.

The tensile engineering stress-strain curves of the four variations of grain morphologies examined in this work are shown in Figure 2a and their corresponding EBSD-inverse pole figure (IPF) maps are shown in Figure 2b. As shown in the figure, there are two homogeneous grain structures (Hom-1 and Hom-2) which were recrystallized at 1100°C for 2 and 1.5 min, respectively. The two heterogeneous lamella structures (HL-1 and HL-2) were achieved by annealing at 975°C for 15 min and 1000°C for 2 min, respectively. The Hom-1 specimen possesses the coarsest microstructure (avg. grain size $\sim 21 \mu\text{m}$) among the four materials and

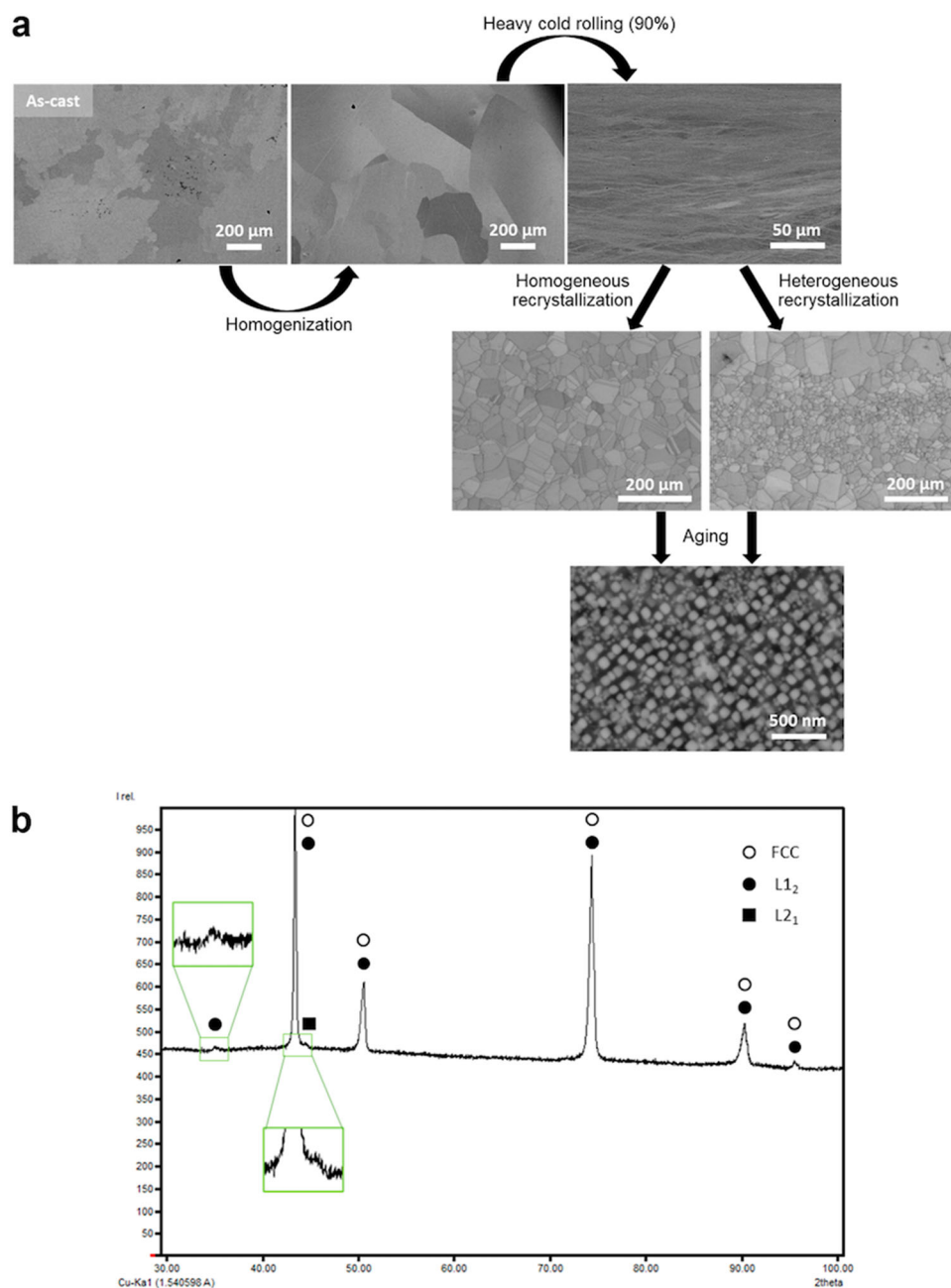


Figure 1. (a) Microstructure evolution of $\text{Al}_5\text{Ti}_7(\text{FeCoNi})_{86}\text{Cu}_2$ through thermo-mechanical processing; (b) Representative XRD spectrum of the final microstructures.

has the lowest strengths (867 ± 4 MPa yield strength and 1289 ± 1 MPa ultimate tensile strength (UTS)). The Hom-2 specimen with finer grains (avg. grain size ~ 17 μm) exhibited increased strengths (924 ± 4 MPa yield strength and 1331 ± 6 MPa UTS) but also a reduction in ductility (from $14.4\% \pm 0.5\%$ to $11.4\% \pm 0.1\%$), which demonstrates the typical strength-ductility trade-off due to grain refinement. On the other hand, the HL-1 specimen, having the almost same yield strength as Hom-2, was able to achieve significantly higher ductility of $16.5\% \pm 0.3\%$ (even surpassed that of the Hom-1

specimen) and higher UTS of 1353 ± 3 MPa benefited from the enhanced strain hardening capability created by the HL structure [30,39]. Similar to HL-1, the HL-2 specimen, which possesses a finer version of HL structure than HL-1, also achieved synergistic enhancements in both strength and ductility (with 1032 ± 2 MPa yield strength, 1411 ± 1 MPa UTS, and $15\% \pm 1\%$ ductility) compared to the two homogeneous-structured specimens.

Similar mechanical behaviors have been reported and explained extensively in multiple other systems with HL structures enabled by grain size heterogeneity

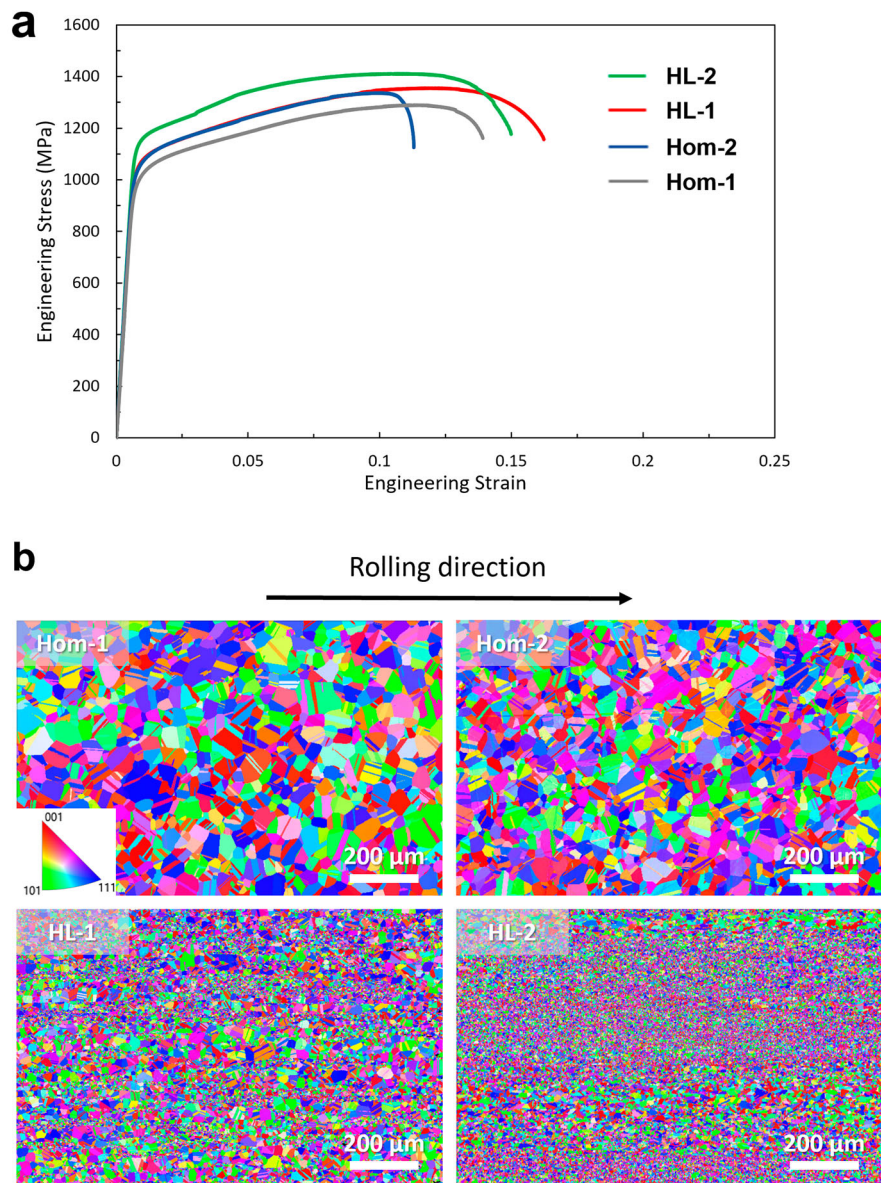


Figure 2. (a) Tensile engineering stress–strain curves of $\text{Al}_5\text{Ti}_7(\text{FeCoNi})_{86}\text{Cu}_2$ with different grain morphologies and (b) their corresponding EBSD-IPF maps.

[7,14,32,35,38–40], where the enhancement in strain hardening capability was attributed to the pile-up of geometrically necessary dislocations (GNDs) during the heterogeneous plastic deformation resulting from different regions having distinctively different grain sizes, and thus, disparate mechanical properties. Whereas in homogenous microstructures, plastic deformation is dominated by the randomly distributed, statistically stored dislocations (SSDs). The accumulated GNDs across the microstructure generate long-range back and forward stresses, collectively defined as HDI stress, which promotes strain hardening and endows the material superior mechanical properties. This special hardening process was termed HDI hardening [31].

HDI stress can be verified by the loading-unloading-reloading (LUR) cyclic tensile test and calculated through interpreting the measured curve [28,31]. Materials with heterogeneous deformation exhibit hysteresis loops in the LUR curve, also known as the Bauschinger effect. Larger loops characterize more substantial HDI hardening [28]. Figure 3a shows the LUR curve generated in the HL-1 material. As can be observed from the curve, the loop becomes progressively larger with the increasing plastic strain, indicating continuously increased HDI hardening effect as the material deforms further. Figure 3b shows calculated HDI stresses at different strain level using the well-recognized method derived by Yang et al. in 2016 [41]. The evolution of HDI stress agrees well

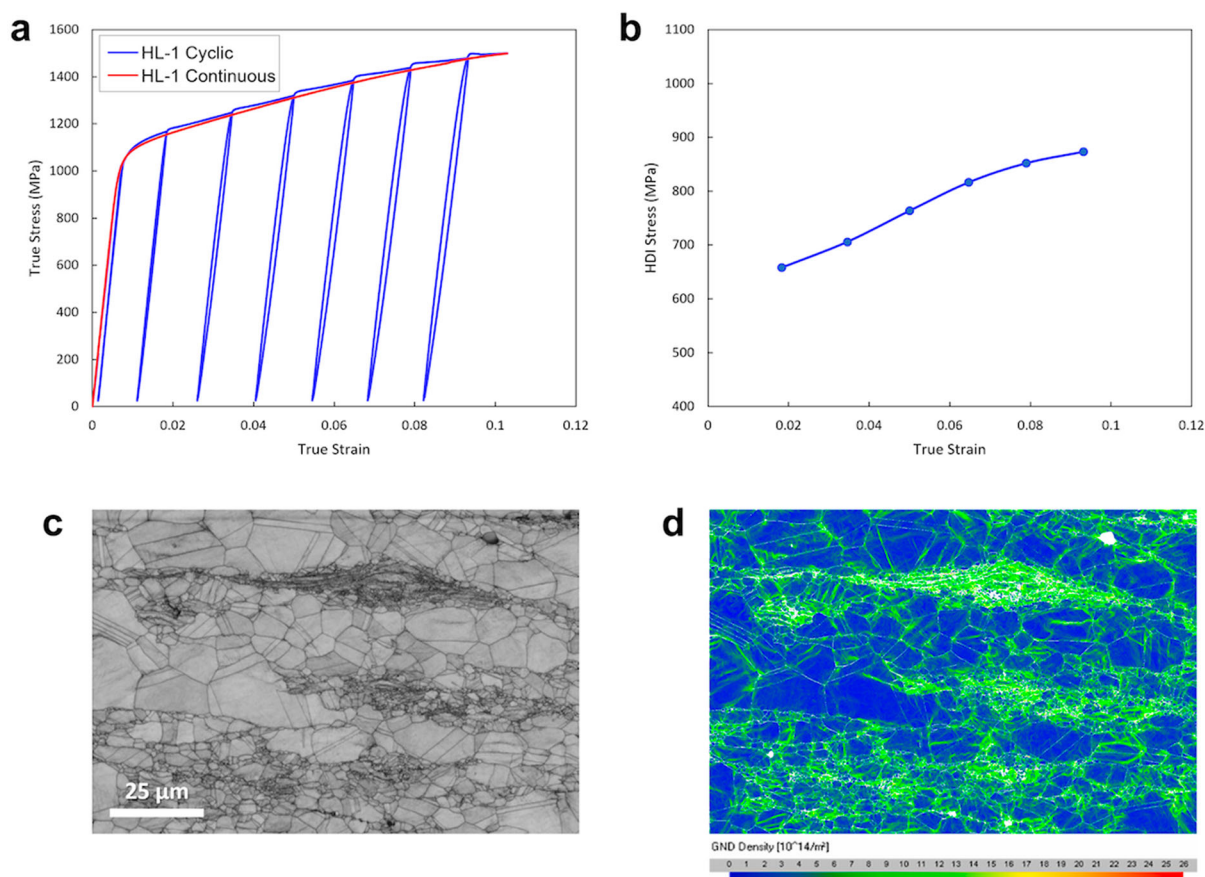


Figure 3. (a) Overlay of tensile true stress–strain curves of the HL-1 specimen under normal continuous loading and cyclic loading; (b) HDI stress calculated from (a); (c) SEM micrograph of deformed HL-1 specimen and (d) its corresponding GND density map.

with the observation from the LUR curve. Figure 3c,d show an SEM image and its corresponding GND density heatmap, respectively, acquired from the deformed specimen. According to the literature, the GNDs primarily forms in the soft, coarse grain region during the yielding stage of the material, which develops back stress in the soft zone and enhances the overall yield strength. Once the material has fully yielded, a forward stress develops in the hard, fine grain region due to the hetero-plastic deformation and is responsible for promoting strain hardening in the hard zone [28]. This forward stress may correlate with the higher GND density observed in the hard zone after plastic deformation. The GND segregation with respect to grain size observed in Figure 3d provides clear evidence of strain partitioning between coarse grain and fine grain regions, which leads to the generation of the HDI hardening effect. The results shown in Figure 3 correspond well with those reported in the literature [7,14,32,35,39,40].

The magnetic hysteresis curves of the four specimens were measured to examine the effect of HL structures on the magnetic performance of materials and are presented in Figure 4. According to the results, all specimens show

typical soft ferromagnetic behaviors, with high saturation magnetization ($\sim 100 \text{ Am}^2 \text{ kg}^{-1}$ on average) and medium coercivity ($\sim 1100 \text{ A/m}$ on average) relative to other FeCoNi-based CCAs (typically $50\text{--}130 \text{ Am}^2 \text{ kg}^{-1}$ for the former and $300\text{--}1500 \text{ A/m}$ for the latter) [25,27,43,44]. As can be further observed in the inset of Figure 4a, both HL-structured specimens achieved higher saturation magnetization than the homogeneous-structured specimens and are less sensitive to the change in grain size. Similarly, both HL-structured specimens exhibited lower coercivity (Figure 4b), with HL-1 performing slightly better than HL-2. It has been previously reported that the magnetic properties of soft magnetic CCAs can be improved by tailoring the chemical composition or microstructure, but the improvements have either been accompanied by compromises in mechanical properties [9,24,26,42] or not been able to be realized synergically (e.g. the improvement in coercivity required sacrifice in saturation magnetization) [11,25,27,43,44]. The results from this study suggest that both properties can be simultaneously enhanced (and each in a synergic way) by utilizing the HL structure with grain size heterogeneity.

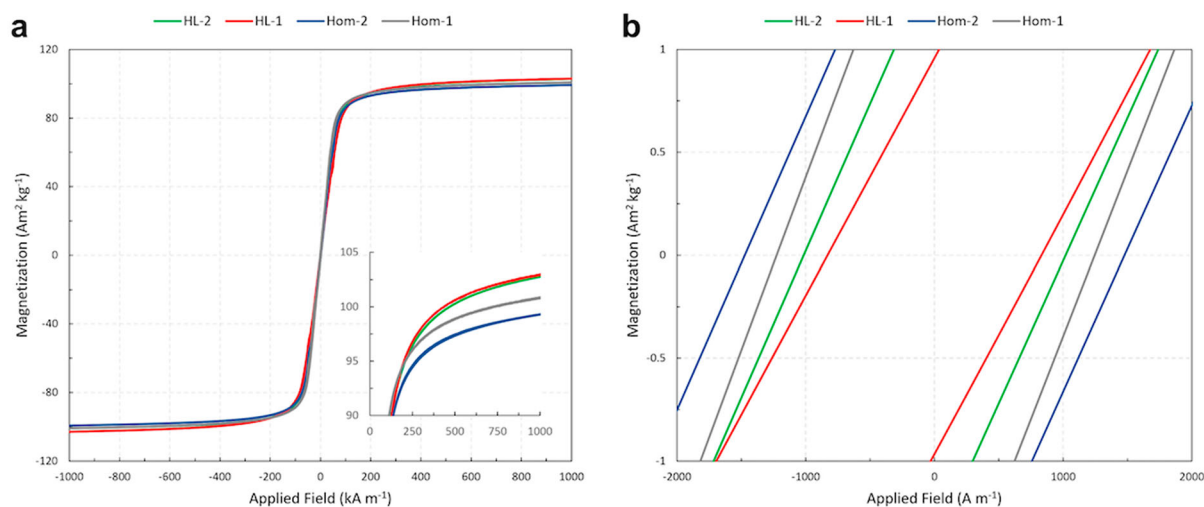


Figure 4. (a) Magnetic hysteresis plots of the four specimens with the inset showing details of saturation behavior; (b) Magnified view on the center region of the plots in (a).

It has been demonstrated that the magnetic properties of superalloy-like CCAs are typically dominated by the composition of the FCC matrix and the L_{12} precipitates [9,11]. In the present study, this factor was controlled by using the same alloy composition and same aging parameters in different specimens. It has also been shown that the magnetic properties can be effectively tuned by adjusting grain size distributions [45]. Therefore, the dominant variable on the magnetic behaviors in the current materials is more likely to be the different grain size distributions achieved in the recrystallization step prior to aging. For instance, in the two homogeneous-structured specimens, the finer grain size of the Hom-2 specimen compared to Hom-1 lead to lower saturation magnetization and higher coercivity due to the increased impediment of grain boundaries on the growth of domain walls [9,11]. However, in the HL-structured specimens that possess a finer microstructure compared to either Hom-1 or Hom-2, improvements in both saturation magnetization and coercivity have been achieved. A plausible explanation to this phenomenon is the synergistic effect produced by the interactive coupling between heterogeneous zones which has been observed in many functional materials including magnetic materials, thermoelectric materials, ferroelectric materials, etc., where integrated properties can exceed properties predicted by rule-of-mixture from separate zones [31]. Such synergies have been achieved in several composite magnetic materials with heterogeneous structures, but have not been reported in precipitation strengthened alloys with heterogeneity enabled by grain size variations. Therefore, more in-detail studies focusing on the magnetic behaviors in materials of this type will need to be carried out to better understand the novel behavior observed in the present work.

Conclusion

In summary, HL structures were produced in a FeCoNi-based soft magnetic superalloy-like CCA via simple thermo-mechanical processing. Compared to the specimens with homogeneous grain structure, the HL-structured specimens exhibited strength-ductility synergy and saturation magnetization-coercivity synergy, both at the same time, which has not been realized in previous studies on soft magnetic CCAs. Furthermore, the production of HL structure in this work do not require special processing steps (e.g. asymmetric rolling, extra hot rolling and/or extra annealing) and can be generalized to many other similar systems, which is particularly beneficial for CCAs that have countless compositional possibilities. Finding a generally applicable processing route for the same type of microstructure can minimize the compositional dependence of performance and significantly reduce the lead time and cost compared to extensive composition mining and validation [46].

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Disclosure statement

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