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Magnetic behavior of nanoporous FeCo, fully and partially dealloyed by liquid metal

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Liquid Metal Dealloying (LMD) is recommended to enhance the near-surface ferromagnetic properties of Iron-Cobalt alloys. With a lower surface electrical conductivity, treated specimens are expected to accommodate the frequency effect better and exhibit a lower level of magnetic loss. This work focuses on the magnetic behavior of LMD-treated samples. The magnetic hysteresis cycles of a virgin, a fully, and a partially dealloyed specimens are plotted at different levels of frequencies of the magnetic excitation. Then, a model to simulate those cycles is described. The simulation parameters of the fully and virgin specimens can be leveraged to predict the behavior of the partially dealloyed specimen and estimate the thickness of the treated area accurately and non-destructively.

Index Terms—Metal dealloying, non-destructive testing, surface treatment, magnetic behavior.

I. INTRODUCTION

LQUID METAL DEALLOYING (LMD) is a metallurgical process that selectively removes components from an alloy, creating a porous structure. The process involves four stages: (i) elaboration of a precursor alloy, (ii) immersion into a liquid metal that selectively dissolves one component, (iii) solidification of the liquid metal leading to the formation of a composite microstructure, (iv) selective etching of one of the phases to obtain porous structures. The dealloying process is versatile, as tuning the parameters (composition of the precursor and of the melt, dealloying time and temperature, etc.) to control the resulting porous microstructure is possible.

LMD finds applications in catalysis, sensing, electrochemical applications, and biomedical devices [1], but its potential in electromagnetic conversion has been underexplored. However, by selectively removing elements, LMD can create nanoporous structures with improved magnetic properties [2].

Permendur, a well-known FeCo alloy, is recommended in the soft ferromagnetic community for its numerous advantages, including high saturation flux density, low coercivity, and reduced core losses. The high saturation level of Permendur allows for smaller and lighter magnetic cores in applications like lightweight aircraft transformers and electric motors [3]. While increasing saturation level enhances power density, higher working frequencies achieve similar results, albeit with increased magnetic losses. The study suggests using LMD to modify FeCoNi alloy plates (with a similar FeCo proportion as Permendur) to create a locally porous structure with reduced electrical conductivity and magnetic losses. The diffusion-based LMD process allows precise control over dealloyed part thickness, balancing magnetic and mechanical properties.

To evaluate LMD outcomes non-destructively, the work describes a simulation method reproducing hysteresis cycles of

virgin and fully dealloyed specimens at different frequencies. The objectives of this work include verifying if dealloying improves magnetic performances, observing and simulating the magnetic dynamic behavior of fully or partially porous materials, and non-destructively determining LMD treatment trajectory in ferromagnetic parts (thickness of the dealloyed part).

The study's significance extends beyond LMD, as the proposed evaluation methods can apply to various metal surface treatments involving diffusion, such as carburization or nitriding in steel parts and silicon diffusion in electrical steel.

II. TESTED SPECIMENS AND MAGNETIC CHARACTERIZATION

In this work, a button-shaped ingot of (FeCo)70Ni30 was prepared using arc melting with high-purity raw materials. The ingot underwent multiple cycles to ensure chemical homogeneity and was cast into three plate samples. These samples were cold-rolled, heat-treated, and used as precursors. The LMD process involved heating a metallic melt containing Mg-10at%Ca in a purified He atmosphere. The process selectively dissolved Ni atoms from the precursor alloys into the melt, while Fe and Co atoms formed a 3D interconnected structure [2]. Mg melt channels also grew into the precursor alloy, resulting in a composite material with interconnected FeCo and Mg phases in a 3D structure. Subsequent etching selectively removed the Mg phases. Three categories of MD-treated specimens were prepared: untreated, treated for 200 seconds, and treated for 2 hours (see right part of Fig. 1).

The process demonstrated the formation of a composite material with a unique 3D structure, showcasing the potential of the LMD process for creating advanced materials. Then, a dedicated testing setup was built to assess the average magnetic flux density (B_a) in response to a tangential surface excitation magnetic field (H_{surf}) at various frequency levels. A picture of this experimental setup is provided in the left part of Fig. 1.

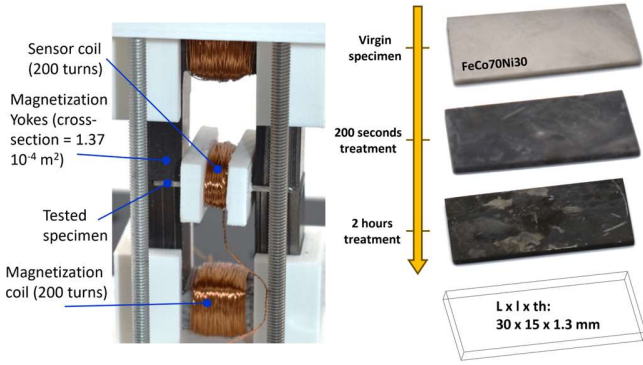


Fig. 1. Left: experimental setup. Right: tested specimens and dimensions.

III. HYSTERESIS SIMULATION METHOD

The simulation process used in this study originates from a method initially introduced in [4]. This technique models the magnetic behavior of a ferromagnetic lamination by simultaneously solving the magnetic field diffusion equation (Eq. 1) and a time-dependent material law (Eq. 2). In the case of a ferromagnetic lamination, where the thickness ζ is reduced compared to both width and length, a one-dimensional (1D) resolution of the diffusion equation is sufficient for obtaining accurate simulation results (Fig. 2, right part):

$$\sigma \frac{\partial B}{\partial t} = \frac{\partial^2 H}{\partial x^2} \quad (1)$$

Here, x is in the thickness direction. A Finite Differences (FD) discretization is typically applied for the resolution. In [5], an extensive frequency range test revealed that a first-order differential equation (analogous to viscous behavior) offers limited accuracy. Eq. 2, incorporating a fractional derivative term, produces much superior results. In this equation, ρ is a constant, and $H_{stat}(B)$ represents the magnetic excitation field required for obtaining magnetic flux density B in the quasi-static regime:

$$\rho \frac{d^n B}{dt^n} = H - H_{stat}(B) \quad (2)$$

As discussed in previous works [6], fractional derivatives are mathematical operators balancing the frequency effect differently from an integer order derivative. It gives flexibility and accurate results across a significant frequency range.

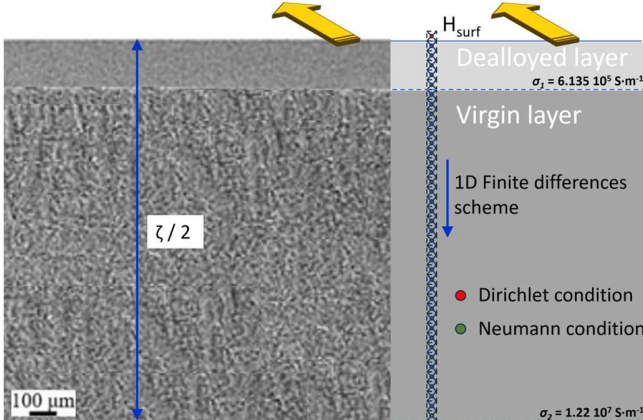


Fig. 2. Left: SEM image of a partially dealloyed specimen. Right: FD discretization.

In the SEM image of a partially dealloyed specimen (Fig. 2), we can observe a significant contrast between the treated and untreated areas. Here, the simulation method is based on this difference. It simulated the dealloyed part using the simulation parameters of the fully dealloyed specimen and the untouched region with the virgin specimen ones. The thickness of the dealloyed part is the only degree of freedom. It is set by an optimization process comparing simulations and measurements (relative Euclidean difference). Accurate simulation results allow us to predict the dealloyed area's thickness precisely.

IV. EXPERIMENTAL AND SIMULATION RESULTS

Fig. 3 below compares the simulated and the experimental hysteresis cycles measured on the partially dealloyed specimen. The simulation method predicted a thickness of 100 μm for the dealloyed part, which was confirmed afterward by SEM images.

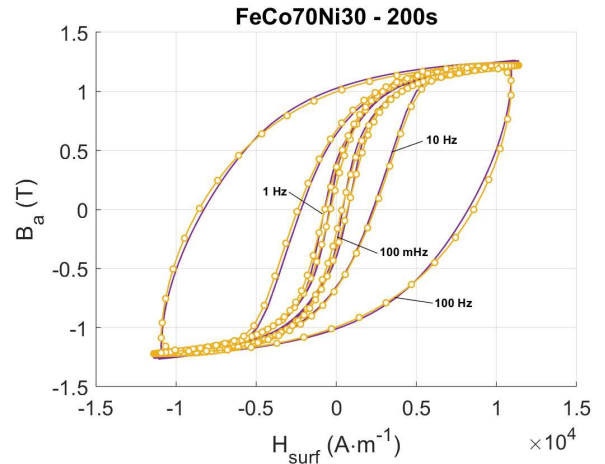


Fig. 3. Comparison simulation/measurement for the partially dealloyed specimen and freq. = 0.1, 1, 10, and 100 Hz.

V. CONCLUSIONS

This work focused on partially dealloyed FeCo specimens. Magnetic tests were done, and a simulation method was described to accurately predict the dealloyed region's thickness non-destructively. Extended descriptions and additional results will be provided in the long-version manuscript.

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