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DIFFERENT INFLUENCE OF SQUEEZING SURFACES ON ANISOTROPY OF RING CORES AND STRIPS OF Fe-BASED NANOCRYSTALLINE RIBBONS

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Soft-magnetic properties of Fe-rich and Si-poor Fe-Nb-Cu-B/P-Si nanocrystalline alloys were investigated. Ar-annealed ribbon-wound ring cores and straight strips were compared and core loss was measured. Although phosphorus was found to hamper preferred surface crystallization - one of the sources of surface squeezing that leads to poorly controlled hard-ribbon axis anisotropy, P appears to aid the other source - oxidation and thus the surface squeezing persists in P-containing alloys too. Whereas P substitution improved slightly the core loss in 4 at% Si alloy, it worsened the loss in 8 at % Si alloy possibly due to making hysteresis loops of ring core and straight strip different as a consequence of different surface stress condition. Si to P balance thus appears significant for a successful hi-B material.

Keywords: magnetic domain structure, nanocrystalline alloys, power loss, soft-magnetic materials

1 INTRODUCTION

Popular Fe-Nb-Cu-B-Si Finemets are already established in industrial use. To get still more from an established material is a standard practice. The demand for mass production based on Finemet-like material is: increase the induction (make hi-B material), lower the price and at least keep the functional (soft-magnetic) parameters. The demand transforms to: increase Fe percentage, spare/avoid Curie-temperature-lowering components (Si, Nb, B) as well as "strategic" and expensive ones (Co, Nb, Mo...). We tried to see what happens to certain functional properties, less common ones (surface properties) inclusive, if we move from standard Finemet composition in the hi-B direction. Attaining true hi-B (over 1.5 T) was not the aim. In this work we kept the grain-growth blocker (Nb) in the basic composition since our previous research [1] found that omitting Nb necessitates the use of fairly limited annealing temperature (390°C to 450°C [2, 3]) and/or duration and thus limits material's thermal stability.

2 EXPERIMENTAL

Amorphous ribbons of composition $Fe_{78-x}Nb_3Cu_1B_{14-y}P_ySi_{4+x}$ (x= 0, 4, y= 0, 3) were prepared by the planar-flow casting on air; labeling Si4, Si4P, Si8 and Si8P. Strips of 10mm width, 10cm long and ring cores (30 mm mean diameter, ~4g mass) were annealed in technical purity Ar ambience at 520°C for 1h to reach nanocrystallization. Hysteresis loops were recorded on digitizing hysteresisgraph at 21 Hz in Helmholtz drive coils along the ribbon long axis. Core loss (ring cores) was evaluated from minor loop area at 5 kHz up to 0.9T. Low output impedance amplifier (no feedback one) was used to excite the cores. However, it was difficult to construct equal more massive (to increase input impedance with modest standard primary) ring cores from brittle ribbons (Si8P) too. Therefore power loss measurements are not performed in pure sin B regime. Electron Dispersive Spectroscopy (EDS) was used to look for differences of element abundance at different locations as well as between surfaces and bulk. Magnetic domain structure (DS) was observed by a digitally enhanced set up that uses magneto-optical Kerr effect (MOKE) and enables subtraction of domain-free (magnetically saturated) surface image. Whereas maximum magneto-optical sensitivity (MMOS) has been aligned horizontally or vertically to distinct domain magnetization direction, external bias field was always vertical (along long ribbon axis) on all the images displayed. All the surface analysis methods were used on both the opposite ribbon surfaces – the air side (AS) and the wheel side (WS).

3 RESULTS AND DISCUSSION

This work focuses on samples annealed in technical purity Ar. This means that additional (superficial) ribbon-surface oxidation is readily expected. There are more (and more pronounced) effects due to composition change (B replaced by P) and/or surface ambience condition (single strip vs ribbon-wound core) if additional oxidation is enabled during nanocrystalllization, thus we can learn more. The most pronounced consequence of B/P substitution revealed after vacuum annealing of Si4P and Si8P is the reversal of slight surface crystallization preference (observed in Si4 and Si8) and suppressed overall

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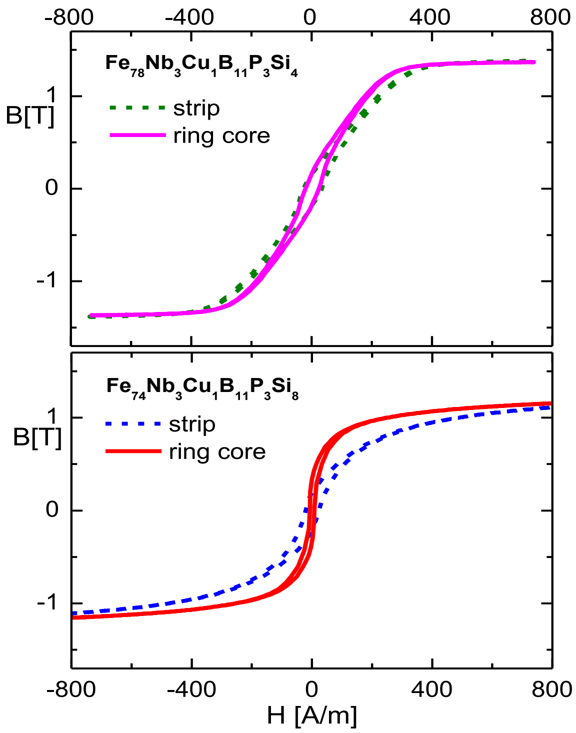

Fig. 1. 21 Hz hysteresis loops of Si4P and Si8P annealed strips and ring cores

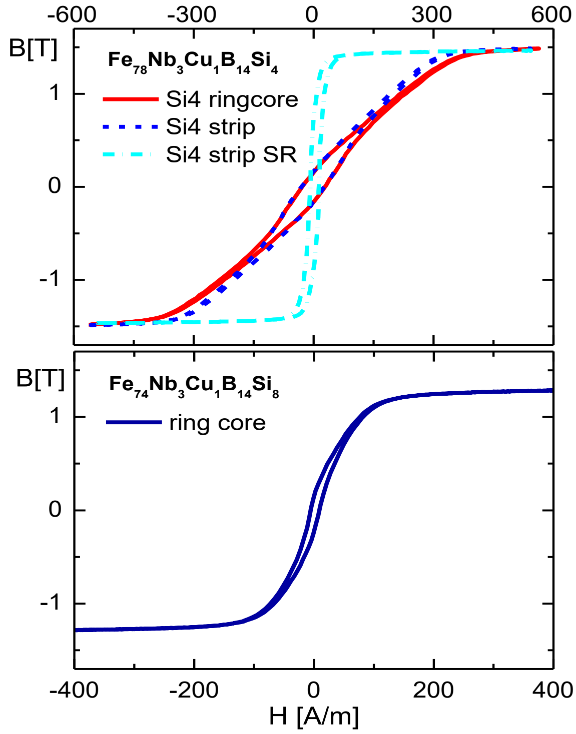

Fig. 2. 21 Hz hysteresis loops of Si4 and Si8 annealed strips and ring cores

crystallinity after equivalent treatment. Analysis of the Ar-annealed as well as as-cast ribbons by EDS showed no notable differences, neither across the width, nor across the thickness of the ribbons.

3.1 Hysteresis loops and surface effect

As a rule, loops of Ar-annealed Si-poor FeNbCuBSi become slant. This might support rotational processes and diminish core loss and flatten its frequency variation. Too much loop tilt comes from too much hard-ribbon-axis anisotropy caused by in-plane compressive stress exerted by surfaces on the ribbon interior and the following hard-ribbon-plane anisotropy creates a loop "belly" as seen in Fig. 1.

The surface stress comes from preferred surface crystallization supported by surface oxidation as shown by upright loop after surface removal (SR) in Fig 2. Since the ambient conditions for the formation of such surface layer can differ between single strip and tightly wound ring core, we compared the response of the sample forms. Indeed, for Si8P (unlike the other compositions) the response is notably different as seen in lower Fig. 1. Apparently two explanations are at hand: (i) Si8P shows higher magnetostriction than Si8 [4] because P reduces the effect of lowering magnetostriction with increasing crystallinity that are observed in P-free similar alloys [5] and making a thin ring core causes tension along ribbon axis to prevail . (ii) AS and WS of Si8P (unlike Si8) differ so that opposite stress on AS to WS helps to diminish the resulting surface stress. Indeed, previous CEMS study [6] revealed much more oxides on WS than on AS of Si8P. There is no notable difference between straight-strip and ring-core loops of Si8 (therefore not both the loops appear on lower Fig. 2) so that we prefer ii) as the more probable explanation.

3.2 Power loss

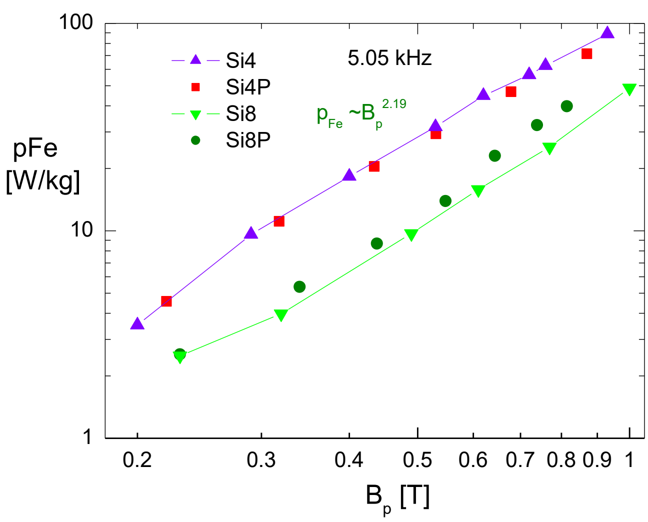

Fig. 3. Power loss of 520°C/1h Ar-annealed ring cores

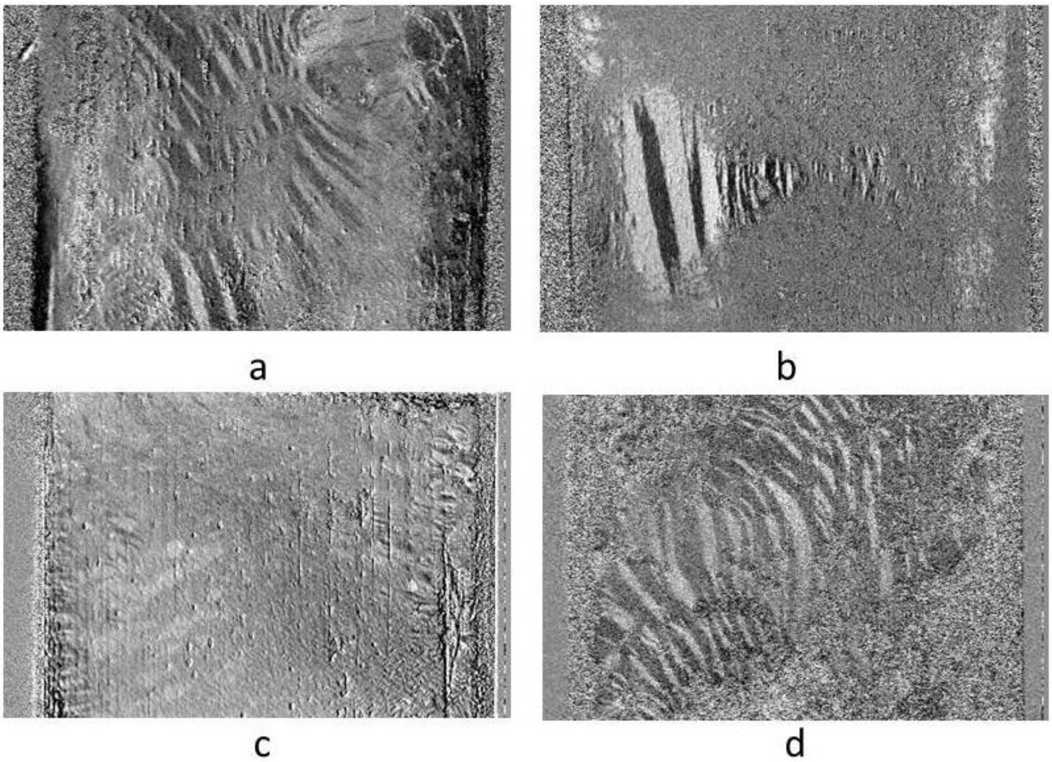

Fig. 4. Domain structures of strips annealed at 520°C/1h in Ar: (a) – AS Si4P, (b) – WS Si4P, (c) – AS Si8P and (d) – WS Si8P whole ribbon width (10 mm) is seen (demagnetized state). Vertical MMOS is used for (a) and (b), horizontal one for (c) and (d).

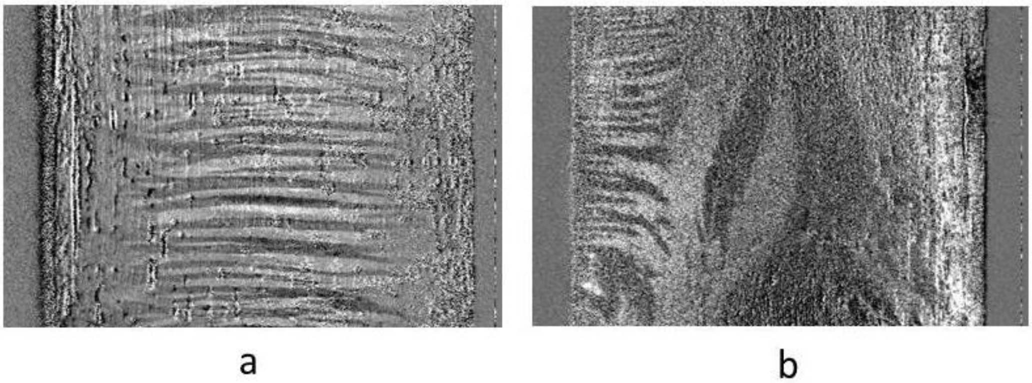

Fig. 5. Domain structures of strips annealed at 520°C/1h in Ar: (a) AS Si8, (b) WS Si8, MMOS is horizontal

The loop shape observed for Si8P ring core could also be the reason for the somewhat higher core loss if compared to Si8 (see Fig. 3) - although showing less coercivity at 21 Hz, the loop shape shows preference for domain-wall movements and steeper p_{Fe}/B-slope. The higher loss for Si4 and Si4P comes mainly from the loop "belly" visible at low fields which in turn points to stronger effect of surface-generated stress on the higher-magnetostriction material [7].

3.3. Domain structure.

Unfortunately the observed domain structure (Fig. 4.) only pertains to straight strips and being layered, the observed structure does not reveal true volume domain structure to support core loss explanation. But compared to Si8 (Fig. 5.) it points to the mentioned difference between AS and WS of Si8P: Whereas Si8 WS on Fig. 5b shows some longitudinal domains magnetized at an angle to the ribbon axis (low contrast) in Fig. 4d, (Si8P) shows closure domains on its WS that point to fairly larger in-plane compressive stress exerted by this surface on the ribbon interior.

Closure domains are revealed by their head-on magnetization [8] and emerge as a consequence of out-of-plane magnetic flux induced by in-plane compression of positively magnetostrictive material beneath.

Worth noting for the Si-poor Fe-Nb-Cu-B-Si alloys is the property of creep-induced-like anisotropy with easy direction parallel to the easy direction produced by magnetoelastic interaction [9]. Whereas Si8 belongs to the group of materials that create no such anisotropy, Si8P does according to preliminary measurements. Thus this anisotropy can add to the magnetoelastic one if the stress conditions do not reverse during cool down of the annealed samples. Summing up, the incorporation of P into composition of Fe-rich and Si-poor alloys brings mixed results and necessitates further research. As for the surface oxidation, it appears that this is no separate source (it was not observed to generate notable stress without surface crystallization) [1] but can support the surface stress together with surface crystallization.

4 CONCLUSIONS

- Thus replacing some B bys P "helps" slightly soft-magnetic properties of Si4 but not those of Si8. The Si4P to Si8P differences point to some sort of Si-P competition
- Alloys Si4 and Si4P show only minute influence due to partial substitution P instead of B as well as only minute difference between straight strip and ring-core loops. Core loss is insignificantly better for Si4P.
- Increasing silicon to Si8 lowers positive magnetostriction and increases effects due to B/P partial substitution. Whereas loops for straight strip and ring core differ minimally for Si8, for Si8P the loop of ring core becomes significantly less slant and the core loss rises compared to Si8.
- Thus substituting B for P "helps" slightly soft-magnetic properties of Si4 but not those of Si8. The Si4P to Si8P differences point to some sort of Si-P competition.

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