

Effect of Surfaces of FeNbCuBSiP Ribbons

P. BUTVIN^{a,*}, B. BUTVINOVÁ^a, M. KUZMINSKI^b, A. ŚLAWSKA-WANIEWSKA^b, J. SITEK^c,
I. MAŤKO^a, M. KADLEČÍKOVÁ^d

^aInstitute of Physics, Slovak Academy of Sciences, Dúbravská cesta 9, 845 11 Bratislava, Slovakia

^bInstitute of Physics, Polish Academy of Sciences, Al. Lotników 32/46, 02-668 Warszawa, Poland

^cInstitute of Nuclear and Physical Engineering, STU, Ilkovičova 3, 812 19 Bratislava, Slovakia

^dInstitute of Electronics and Photonics FEI, STU, Ilkovičova 3, 812 19 Bratislava, Slovakia

Boron was partially substituted by phosphorus (3 at.%) in two Si-poor Fe-Nb-Cu-B-Si Finemets. Mostly non-significant changes were observed after vacuum annealing at 500 °C, whereas equivalent Ar annealing resulted in significantly better soft-magnetic properties, which suffer from compressive surfaces stress. Possibly by hampering surface crystallization, the substitution eased the surfaces stress and reduced undesired off-axis anisotropy at the cost of limiting the useful annealing temperature.

DOI: [10.12693/APhysPolA.126.152](https://doi.org/10.12693/APhysPolA.126.152)

PACS: 75.30.Gw, 75.50.Tt, 75.60.Ch

1. Introduction

Si-poor Finemets show good soft-magnetic properties with significantly higher saturation than “classic” Si-rich compositions. The good properties deteriorate during non-vacuum annealing due to stress exerted by surfaces (crystallizing first) on the ribbon interior. Thus we tried to influence the surfaces by substituting a part of boron by phosphorus in a composition between Si-poor and practically Nb, Cu and Si-free one [1].

2. Materials and methods

Metallic glass precursor ribbons of Fe₇₈ or Fe₇₄Nb₃Cu₁B₁₀P₃Si₅ or 9 at.% were prepared by the planar-flow casting on air. Strips of 10 mm width and 20–23 μm thickness were annealed in vacuum or in Ar ambience at 500 °C for 1 h. Sample contamination (fingerprints etc.) has been carefully avoided, but no specific surface cleaning was attempted. Hysteresis loops were recorded using a digitizing hysteresisgraph at 21 Hz sinusoidal H excitation in Helmholtz drive coils, along the ribbon long axis. Magnetic domains were observed by Kerr-effect method (MOKE). Structure, critical temperatures (T_x , T_C) and magnetostriction were studied previously [2] and are augmented by Mössbauer spectroscopy (MS) in this work. Electron Dispersive Spectroscopy (EDS) was used to look over differences of element composition between surfaces and bulk. Investigation of surface chemistry of the strips was performed by Raman spectroscopy (RS) using He-Ne laser (632.8 nm).

3. Results and discussion

If compared to non-substituted composition, vacuum-annealed Si₅ shows a significantly reduced coercivity by 60% (attaining 6.7 A/m) but also a slightly lower saturation (1.38 T). The substitution did not improve the coercivity in Si₉ but increased saturation to 1.36 T. This

is due to increased Curie temperature – the explanation of this somewhat surprising change will be sought in near future. Otherwise, the upright loop shape after vacuum annealing does not reflect the substitution, whose effect is far more pronounced after Ar annealing, indicating a change of the surface properties. Whereas Si₅ continues to show reduced transverse anisotropy after Ar-annealing at even higher temperature, 500 °C is the upper limit for Si₉ to show properties improved by the substitution. Figure 1 shows that annealing at higher temperature, by even 20 °C, causes 4-fold increase of magnetization work to 41 J/m³ and 25% increase of coercivity (from 16 to 20 A/m).

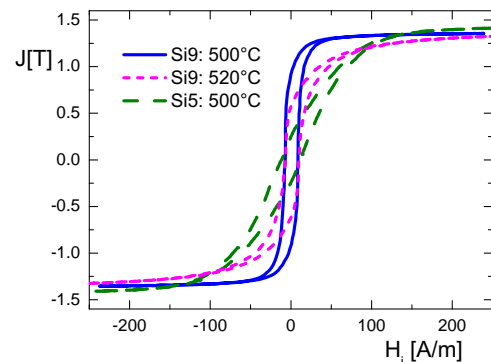


Fig. 1. 21 Hz loops of Ar-annealed strips.

Since loops corresponding to Fig. 2a are quite upright, they imply that the transversal domains are the minor ones. Since the domains are not magnetized “head-on”, they are not the closure domains but stripe domains [3] (of a primary surface structure) and are not considered to fill the ribbon interior. Otherwise, Fig. 2a represents an indication of transversal macroscopic heterogeneity (TMH), which is typical for wider Finemet rib-

*corresponding author; e-mail: pavol.butvin@savba.sk

bons [4]. Whereas longitudinal surface anisotropy prevails at the edges, the central part of the surface is governed by transverse anisotropy due to prevailing tension along ribbon axis and across the ribbon axis respectively. The heterogeneous anisotropy comes from magnetoelastic interaction in Si9 because this silicon percentage in Finemets does not support a significant creep-induced-like anisotropy [5]. The opposite surface (wheel side) displays complementary anisotropy (not shown for Si9, but similar to Fig. 2c), which points to mutually compensated stress on the surfaces. Fig. 2b shows the surface anisotropy and thus TMH starting to decline by decreasing transverse stress during surface crystallization (see Fig. 3 – MS).

Vacuum-annealed Si5 also shows TMH, however with principally similar domain structure on both surfaces. This property could be accounted for by assuming that surfaces stress does not compete with each other, but both surfaces compete and establish a balance with the ribbon interior. The advancing surface crystallization in Ar again appears to reduce TMH but unlike Si9, it does not preserve the majority of longitudinal anisotropy. The slant loop of Si5 (Fig. 1) indicates a transverse anisotropy component, as if the two opposite surfaces compress the ribbon interior in a similar manner. Surface removal by etching lets the loop to show significantly reduced tilt and magnetization work (i.e. weaker transverse anisotropy).

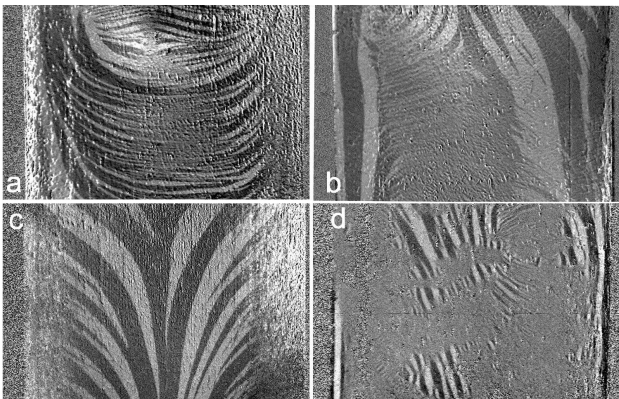


Fig. 2. Domain structures of strips annealed at 500 °C: (a) Si9 in vacuum, (b) Si9 in Ar, (c) Si5 in vacuum and (d) Si5 in Ar. Whole ribbon width is seen (demagnetized state). Wheel side is shown on (c), air side otherwise; horizontal magneto-optical sensitivity is used for (a), vertical one otherwise.

EDS proved that there is no appreciable difference in P percentage between surfaces and interior. It confirmed still extensive oxidation of Ar-annealed surfaces. Oxides do not press, but appear to induce – promote surface crystallization (Fig. 3). The oxides identified by RS are mostly iron oxides (Fe_2O_3 , Fe_3O_4). Clear identification of a P compound was not successful, either. Whereas vacuum annealing of Si9 did not result in well resolved MS subspectra (too weak signal from crystalline-phase

Zeeman splitting), Ar annealing lets partly resolved DO3 superstructure (the four minor subspectra occupying 20% of spectrum area in Fig. 3) to start forming. There is not enough Si to form perfect DO3. The still Si-poorer Si5 shows larger crystalline share of solid solution of Si in Fe for Ar-annealing (40% for Ar, 25% for vacuum). The difference between vacuum and Ar annealing points to surface crystallization.

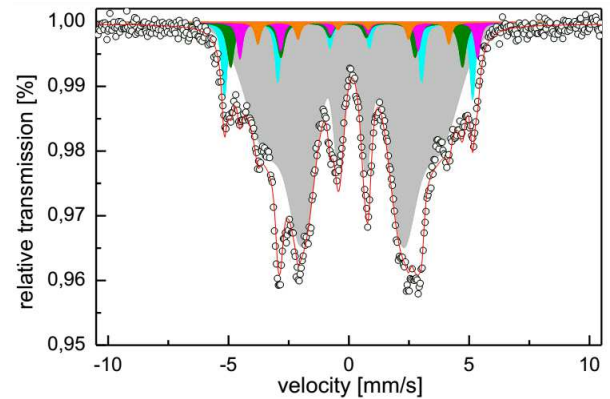


Fig. 3. Mössbauer spectra of Si9 annealed in Ar at 500 °C. The largest-area subspectrum (light gray) belongs to the amorphous rest.

4. Conclusions

Although the substitution of 3 at.% of B by P affects the intrinsic material parameters just slightly and only minor differences are observed after vacuum annealing at 500 °C, it markedly influences surface properties, particularly in ribbons annealed in Ar at 500 °C. Then, the surface action on the ribbon interior remains squeezing, but the compressive stress is significantly reduced by the substitution. This makes the loops less slant, more linear and reduces the coercivity although the magnetostriction is not reduced.

Acknowledgments

This work was partly financial supported by National Agency VEGA projects No. 2/0056/11, 2/0056/12 and 2/0111/11. Authors are also grateful to Center of Excellence FUN-MAT.

References

- [1] A. Urata, H. Matsumoto, S. Yoshida, A. Makino, *IEEE Trans. Mag.* **47**, 3177 (2011).
- [2] B. Butvinová, P. Butvin, E. Illeková, P. Švec Sr., G. Vlasák, D. Janičkovič in: Proc. Sci. Conf. *Physics of Materials 2012* Eds. J. Tóthová, V. Lisý, FEEL, TU Košice 2012, p. 175.
- [3] A. Hubert, R. Schäfer, *Magnetic Domains*, Springer, Berlin 1998.
- [4] B. Butvinová, P. Butvin, M. Kuzminski, M. Kadlečiková, A. Ślawska-Waniewska, *IEEE Trans. Mag.* **48**, 1340 (2012).
- [5] G. Herzer, *IEEE Trans. Mag.* **30**, 4800 (1994).