Morphology, structure and first order reversal curve (*FORC*) studies of nickel nanoparticles electrodeposited on TiO_2 nanotube arrays

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In the past few years, porous metal oxide films such as TiO_2 have been fabricated via self-organizing anodic oxidation for different applications. Hybrid magnetic/semiconducting materials have interesting properties for spintronic applications. We have recently reported electrodeposition of fine nickel particles on TiO_2 nanotubes [1]. In current report, we have investigated magnetic properties of nickel nanoparticles electrodeposited on TiO_2 nanotubes by different techniques including direct current (DC), pulse-current (PC) and cyclic voltammetry deposition (CVD). We observed that each technique provides different morphologies from fine individual particles to large and coalesced nickel. The variation of coercivity and saturation magnetization values recorded is consistent with our observations of different morphologies of nickel particles, as shown in Table 1. Owing to large number of nanoparticles with size and shape varieties first order reversal curve (FORC) studies ere employed. As seen in Fig.1, FORC diagrams for two samples are completely different. In Fig. 1a Ni nanoparticles mainly behave as an ensemble of superparamagnetic and single-domain ferromagnetic particles with the weak inter-particle coupling. In Fig. 1b, one can see two peaks on the ridge along H_c axis. The first one at H_c=81.5 Oe has the largest intensity indicating that the main part of Ni nanoparticles has near the same size and they are at single-domain state with the average dipolar coupling ΔH=54 Oe. The second peak lies in the high coercivity area of the diagram (H_c=423 Oe) and it can be attributed to the relatively large clusters at multidomain state formed by joint nanoparticles. The long tail along H_c axis is an evidence of the wide dispersion of the clusters size, but from the major hysteresis loops this fact is invisible.

Table 1. Magnetic properties of Ni electrodeposited on TiO2 nanotubes by different techniques

Deposition technique	H_c (Θ=0°), Oe	H_c (Θ=90°), Oe	Ms (m.emu)	M_r / M_s (Θ=0°)	M_r / M_s (Θ=90°)
DC electrodeposition	100.02	150.02	0.3	0.18	0.16
Pulse electrodeposition	150.01	150.02	0.35	0.14	0.14
Cyclic voltammetry deposition	200.04	300.03	10.4	0.29	0.14

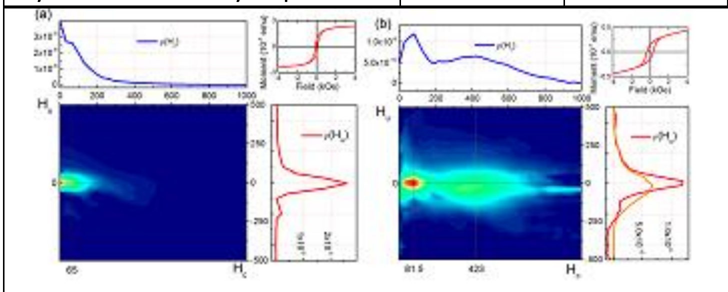

Figure 1. In-plane FORC diagrams for (a) fine indivudal Ni particles and (b) Each diagram is accompanied by the ridge profiles along H_c and H_u axes. Insets consist of in-plane major hysteresis loops.

[1] H. Cheshideh, F. Nasirpouri, J. Electroanal. Chem, 2017, 797, 121-133.