

[0067] At -0.850 V SCE, shown in row (a) of FIG. 4, the deposition potential was so low that the transition to the activated surface 300 and the MBIS passivated one is substantially negligible. Deposition on the trench pattern was limited, but preceded conformally, and the roughness that developed on the sidewalls 308 of the finest feature in column (4) of FIG. 4 lead to incipient occlusion of voids 315 along the centerline of the finest features, as shown in row (a), column (4) of FIG. 4. Increasing the overpotential by 0.025 V to -0.875 V SCE placed the system within the positive feedback hysteretic voltammetric regime. Void-free filling of the finest features was shown along with the development of sloping sidewalls 308 in the wider trenches, as shown in row (b) of FIG. 4. When the overpotential is increased further by 0.025 V to -0.900 V SCE, sloping sidewalls 317 are readily evident in the widest trench shown in row (c), column (1) of FIG. 4 while the remaining trenches are filled and void-free, as shown in row (c), columns (2)-(4) of FIG. 4. For growth at -0.925 V SCE and -0.950 V SCE, all features are shown to be void-free and the overburden thickness 313 increased sharply as shown in rows (d) and (e) of FIG. 4.

[0068] FIG. 5 shows a FIB-TEM cross sectional image of a 160 nm width trench filled by Ni electrodeposition in the presence of 100 $\mu\text{mol/L}$ MBIS where the deposition was performed at -0.925 V SCE for 150 seconds. FIG. 5 shows that the addition of MBIS enables void-free trench filling. FIG. 5 shows that the volume of materials associated with the free surface 404 between the trenches 401 is mostly Cu 403, which is only covered with a thin 25 nm thick Ni layer 405. In contrast, the 160 nm wide trench 401 is entirely filled with Ni 402 and free of any obvious voids. A microstructural seam 407 may exist along the top two-thirds of the trench. This seam 407 or consolidated boundary may be formed during deposition in a manner analogous to grain boundary generation associated with grain coalescence during Volmer-Weber film growth. The bright-field contrast shows an average grain size on the order of about 30 - 40 nm, although certain grains are shown to be larger with well-defined twins or stacking faults evident.

Example 2

[0069] Void-free Ni—Fe deposition was shown onto a dielectric substrate 500 is shown in FIGS. 6(a)-(e). The extension and efficacy of MBIS for inducing superconformal deposition of Ni—Fe alloys is shown. Permalloy, a $\text{Ni}_{80}\text{Fe}_{20}$ alloy that has a rich history in the development of magnetic storage devices was electrodeposited onto a dielectric substrate 500 with the process of Example 1. The electrolytic bath comprised 1 mol/L $\text{NiSO}_4 \cdot 6\text{H}_2\text{O} + 0.2$ mol/L $\text{NiCl}_2 \cdot 6\text{H}_2\text{O} + 0.05$ mol/L $\text{FeSO}_4 + 0.5$ mol/L H_3BO_3 . 100 $\mu\text{mol/L}$ of MBIS was added to the solution and the Ni—Fe alloy was electrodeposited onto the 3-D seeded surfaces of the dielectric substrates with an electric potential of about -0.950 V (SCE) for about 300 seconds. Cross-sectional FESEM images of various patterned trenches showing the deposition of the Ni—Fe alloy 502 are shown in FIGS. 6(a)-(e). Void-free feature filling is clearly shown in each pattern in FIGS. 6(a)-(e). The presence of iron in the electrolytic solution showed a decrease in the deposition rate as compared to that of Example 1. The magnetic properties of the Ni—Fe alloy films were shown not to be significantly altered by MBIS additions in the plating bath.

Example 3

[0070] Void-free Co deposition was shown onto a patterned substrate. The effect of MBIS for inducing void-free deposi-

tion of cobalt is shown in FIGS. 7-9. Cobalt was electrodeposited onto a dielectric substrate with the process of Example 1. The electrolytic bath comprised 0.4 mol/L $\text{CoSO}_4 \cdot 7\text{H}_2\text{O} + 0.01$ mol/L $\text{CoCl}_2 + 0.5$ mol/L H_3BO_3 . Various amounts of MBIS were added to the solution and the Co was electrodeposited onto the 3-D surfaces of the substrates with various electric potentials for various periods of time.

[0071] FIG. 7 shows cross-sectional FESEM images of various patterned trenches showing the deposition of Co 602 in an additive-free deposition at -0.86 V for 300 seconds in rows (a) and (b) for various trench widths, ranging between 720 and 90 nm. Rows (c) and (d) show the deposition of cobalt 602 with an electrolytic solution comprising 200 $\mu\text{mol/L}$ MBIS at -0.86 V for 300 seconds for various trench widths, ranging between 720 and 90 nm. The smaller trenches in the additive-free deposition have void spaces 615, as shown in row (a) column (3) and row (b) columns (1)-(3), of FIG. 7. This is contrasted with the void-free deposition of Co 602 shown in each trench in rows (c) and (d), having MBIS.

[0072] FIG. 8 shows the deposition of cobalt 602 with an electrolytic solution comprising 200 $\mu\text{mol/L}$ MBIS at -0.87 V for 25 seconds in column (1), 50 seconds in column (2), 100 seconds in column (3), 180 seconds in column (4), and 300 seconds in column (5) for various trench widths in each row (a)-(e). The trench widths in FIG. 8 are as follows; row (a) had a trench width of 720 nm, row (b) had a trench width of 280 nm, row (c) had a trench width of 215 nm, row (d) had a trench width of 200 nm, and row (e) had a trench width of 140 nm. Void free deposition of Co 602 in a range of trench widths was shown. In each pattern shown in rows (a)-(e), superconformal filling of Co 602 was exhibited as shown.

[0073] FIG. 9 shows TEM for Co 602 filling using the process of Example 1. The electrolytic bath comprised 200 $\mu\text{mol/L}$ MBIS and the Co 602 was deposited onto the dielectric substrate 601 at an overpotential of -0.86 V SCE. The TEM shows that the micro-structure is void-free with a low density grain boundary marking the 603 center line.

Example 4

[0074] Void-free Co—Fe deposition was shown onto a patterned substrates. FIGS. 10(a)-(f) show $\text{Co}_{80}\text{Fe}_{20}$ 702 trench filling using the process of Example 1 but in a base electrolyte of 0.4 mol/L $\text{CoSO}_4 \cdot 7\text{H}_2\text{O} + 0.01$ mol/L $\text{CoCl}_2 + 0.5$ mol/L $\text{H}_3\text{BO}_3 + 0.01$ mol/L FeSO_4 . The electrolytic bath contained 200 $\mu\text{mol/L}$ MBIS and the alloy was deposited onto the patterned dielectric substrates 703 at an overpotential of -0.87 V for 300 seconds. The patterns varied, having trench widths between 726 nm in FIG. 10(a) and 134 nm in FIG. 10(f). A superconformal void-free deposition of $\text{Co}_{80}\text{Fe}_{20}$ 702 was shown in each of the patterns of FIG. 10(f).

[0075] In each of the Examples 1-4, the addition of MBIS to the electrolytic solution yielded a smoother outer surface with little or no measurable effect on the saturated magnetization (Ms) of the ferromagnetic materials deposited onto the dielectric substrate.

[0076] Examples 1-4 show superconformal electrodeposition of Ni, Ni—Fe, Co, and Co—Fe alloys using a single benzimidazole derivative, MBIS, as an additive to the respective sulfate/chloride mixed electrolyte. The process may offer integration of the ferromagnetic materials into Damascene processes, microelectromechanical systems and related thin-film derived technologies. Feature filling at potentials within the voltammetrically identified critical regimes resulted in void-free superconformal film growth. For freshly immersed