Perpendicular patterned media in an $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs substrate for magnetic storage

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By using electron beam lithography, chemically assisted ion beam etching, and electroplating, we have fabricated high aspect ratio magnetic columns, 60–170 nm in diameter, embedded in an aluminum–gallium–oxide/gallium–arsenide $[(Al_{0.9}Ga_{0.1})_2O_3/GaAs]$ substrate. In our previous work, we demonstrated storage of data in individual columns spaced 2 μm apart. Here the electroplated Ni columns are in the form of tracks (0.5 and 0.25 μm in the down-track direction, and 1 μm in the cross-track direction), corresponding to areal densities of 1.3 and 2.6 Gbits/in.^{2}, respectively. In this report we describe in more detail the issues in the fabrication of patterned media samples, such as dry etching and oxidation of AlGaAs, and electrodeposition of Ni into GaAs substrate. Initial characterization of the resulting magnets using magnetic force microscopy are also presented. [S0734-211X(99)06906-1]

I. INTRODUCTION

Lithographic patterning of arrays of individual nanoscale magnets holds the prospect of advancing magnetic storage to areal densities exceeding the predicted limit of conventionally sputtered, multialloyed thin film recording media. Instead of having hundreds of magnetic grains per bit, patterned media utilizes only one larger-sized magnetic particle for every bit of stored information. Due to the larger volume of each bit, the onset of the superparamagnetic effect, a state at which the individual bit is no longer stable against thermally activated magnetization reversal, should in principle be delayed. Assuming that each bit is small enough to be single domain, and large enough to be thermally stable, a square array of nanomagnets with periodicity of 80 nm or smaller would correspond to storage densities of 100 Gbits/in.^{2} and beyond.

In our initial work, we have focused on creating free-standing magnets of 20 nm diameter and 100 nm spacing (representing a particle density of 65 Gbits/in.^{2}) using electron beam lithography.[1] More realistic structures, made of embedded Ni columns in a durable SiO_2 substrate, to demonstrate the feasibility of using conventional magnetoresistive (MR) sensors to detect the fields coming out of a patterned media sample have also been reported.[2] Previous studies, however, have shown that the coercivity of the resulting columns was not high enough to achieve the magnetic stability required for reliable reading and writing of this form of media.[3]

In order to improve the coercivity of the individual magnetic columns without switching to a different magnetic material system such as cobalt, we have re-examined the fabrication process, and have developed an alternative approach for creating high aspect ratio Ni magnets that are embedded in an Al_2O_3/GaAs substrate.[4] In this approach, we take advantage of the shape anisotropy of the Ni columns to achieve the stability required for the media to be useful for information storage. It has been found that the etch rate selectivity of Al_2O_3 over GaAs can exceed 30:1.[5] Thus, changing to the AlAs/GaAs material system should promote the fabrication of high aspect magnetic columns. The individual magnetic columns, isolated from one another by the nonmagnetic semiconductor substrate, are oriented perpendicular to the surface. Thus, each column represents a ''0'' or ''1'' when its magnetization is oriented either parallel or antiparallel to the long axis of the particle. The columns reported in Ref. 4 are 60–230 nm in diameter, and have a 6:1 aspect ratio. The magnets are arranged in a square array with a 2 μm column-to-column spacing. This configuration was used for the initial recording demonstrations using current read/write technology. We have successfully shown that the magnets made in the Al_2O_3/GaAs matrix are stable against switching when read by a MR element.[4] We have further demonstrated data storage in these individual single domain magnetic nanocolumns (170 nm diameter) using current magnetic recording technology.[6]

Following this proof-of-concept demonstration, we are now working towards making patterned media with columns that are spaced closer together, and still achieving reliable reading and writing of individual columns using conventional read/write technology. Since the read element (MR or spin valve) is much wider in the cross-track direction than it is in the down-track direction, our current work focuses on the fabrication and characterization of patterned media in the form of tracks. These structures have the columns separated in the *x* direction by 1 μm due to the width of the recently obtained spin-valve sensors. The columns are spaced in the *y* direction by 0.5, 0.25, and 0.125 μm in order to allow a

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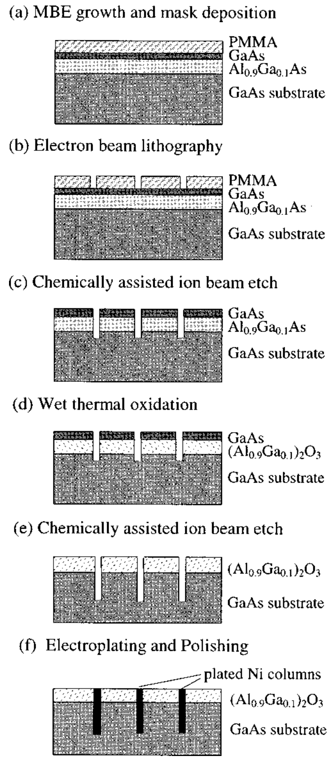


FIG. 1. Schematic diagram of the fabrication procedure of high aspect ratio Ni columns embedded in an $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs substrate.

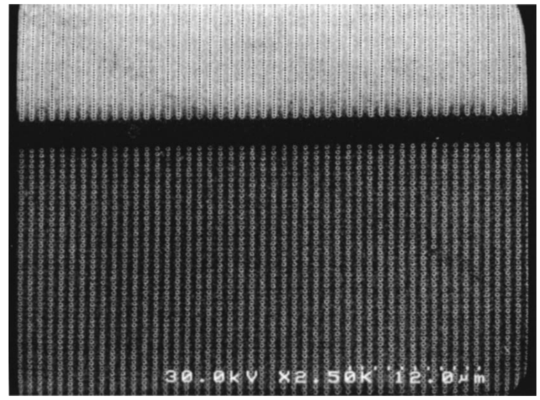


FIG. 2. SEM image of two arrays of holes of 150 nm diameter defined in an $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs substrate. The holes are 0.25 and 0.5 μm apart (top and bottom array, respectively) in the down-track direction, and 1 μm apart in the cross-track direction.

systematic study of the recording properties of patterned media samples with increasing down-track densities. These correspond to areal densities of 1.3, 2.6, and 5.2 Gbits/in.^{2}, respectively. We emphasize that the spacing listed earlier is limited by the widths of the sensors used in our work, and not by our lithographic method, which is capable of defining structures far beyond 50 Gbits/in.^{2}.[1]

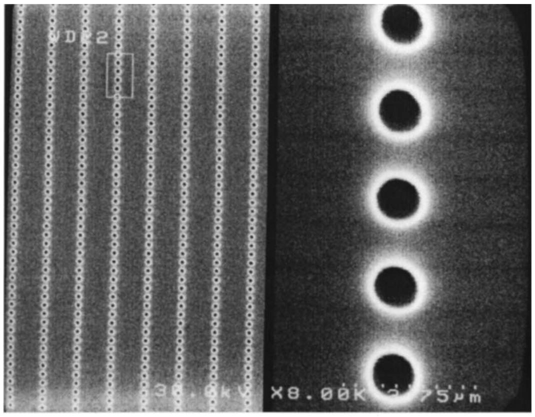
II. PROCEDURE

The fabrication procedure is shown schematically in Fig. 1. A 250 nm layer of $Al_{0.9}Ga_{0.1}As$ and a 100 nm GaAs cap layer are grown by metal-organic chemical vapor deposition (MOCVD) on top of a conductive GaAs substrate. A 15 nm graded layer of AlGaAs is added to both sides of the $Al_{0.9}Ga_{0.1}As$ layer to promote adhesion during the post-etching oxidation process. Instead of using Cr/Au and SiO_2 as etch masks,[4] we now employ only a thicker layer (550 nm) of high molecular weight (950 000) polymethylmethacrylate (PMMA), which serves as both an *e*-beam sensitive resist and an ion etch mask [Fig. 1(a)]. This is a simpler fabrication procedure that still allows us to achieve the required high aspect ratio columns. The dot array patterns are defined on the PMMA (baked at 150 °C for $1\frac{1}{2}$ h) coated sample by vector-scanned electron beam lithography, followed by development in a 3:7 cellulose-methanol mixture [Fig. 1(b)]. The patterns are then selectively transferred into both the GaAs cap and the underlying $Al_{0.9}Ga_{0.1}As$ layer by Cl_2 assisted ion beam etching [Fig. 1(c)].

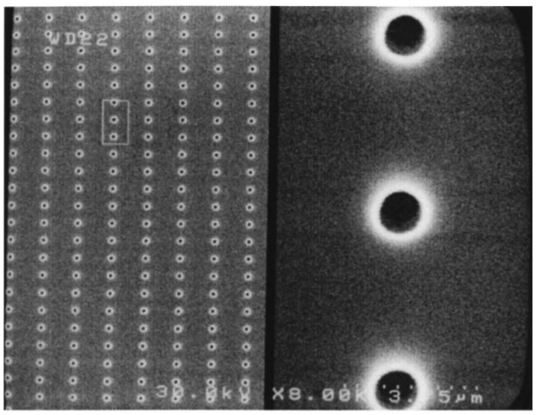
In our chemically assisted ion beam etching (CAIBE) system, a Kauffman Ar^+ ion source is used in conjunction with a gas introduction nozzle to accelerate high energy ions towards the substrate covered with an etch mask. This allows us to achieve a high etching rate and selectivity of the semiconductor substrate, as well as the directionality necessary for defining high aspect ratio structures.

Following CAIBE, the sample is rinsed in acetone and dichloromethane to remove the remaining PMMA. To ensure complete removal of the PMMA after etching, crosslinking of PMMA is avoided by using a lower beam current (20 mA) and keeping the sample stage cooled during CAIBE. The $Al_{0.9}Ga_{0.1}As$ layer is subsequently converted into $(Al_{0.9}Ga_{0.1})_2O_3$ by wet thermal oxidation in a 1 in.-diameter tube furnace at 380 °C for $1\frac{1}{2}$ h [Fig. 1(d)].[7] Water vapor is supplied to the tube by bubbling nitrogen gas at 0.5 liter/min through deionized water heated at 85 °C. To enable a more reproducible oxidation process, each sample is etched right before the oxidation, and nitrogen gas and water vapor is allowed into the furnace $\frac{1}{2}$ h prior to each run. The resulting robust layer of $(Al_{0.9}Ga_{0.1})_2O_3$ is the final mask for pattern amplification in the perpendicular direction into the GaAs substrate by using further CAIBE [Fig. 1(e)]. Figure 2 shows a scanning electron microscopy (SEM) image of two arrays of holes of 150 nm diameter defined in the $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs substrate.

Once the hole array patterns are transferred to the desired depth into the GaAs substrate after additional CAIBE, electroplating is then used to deposit Ni into the holes [Fig. 1(f)]. The plating apparatus is identical to that in Ref. 8, where the probe contacts the sample outside of the plating bath and does not disturb the electrodeposition of the Ni columns. In our case, Ni is used as the anode and the conductive GaAs substrate is used as the cathode. The Ni anode is etched in HCl immediately before each plating session so as to minimize any contaminants to be deposited into the hole arrays. Nickel sulfamate is used as the plating medium and a pulse current with a duty cycle of 80% (2 s on and 0.5 s off) is applied. The plating is conducted under an optical micro-



(a)



(b)

FIG. 3. SEM images of arrays of etched holes in the GaAs cap/ $(Al_{0.9}Ga_{0.1})_2O_3$ /GaAs substrate after CAIBE and oxidation (left). The holes are 120 nm in diameter, with a 0.25 (a) and 0.5 μm (b) spacing in the down-track direction and a 1 μm spacing in the cross-track direction. The right portion of each figure represents a 10× enlargement of the etched holes on the left.

scope, and end point detection is possible through the change in optical contrast of the plated Ni columns. Precise end point detection is not critical in our case as any overplated Ni column can be polished to the desired height, which is determined by the thickness of the $(Al_{0.9}Ga_{0.1})_2O_3$ layer, using a chemical mechanical polish (CMP). For polishing, we use a CMP pad SIP2000A in combination with a polishing slurry which consists of a liquid dispersion of 20–30 nm colloidal silica spheres at a concentration of 28%, and with a pH of 11.3.[9]

III. RESULTS AND DISCUSSION

A. Two-dimensional masking

Using this procedure, we have fabricated arrays of Ni columns that are embedded in a robust $(Al_{0.9}Ga_{0.1})_2O_3$ /GaAs substrate. Figures 3(a) and 3(b) show SEM images of arrays of etched holes, 120 nm in diameter, in the GaAs cap/$(Al_{0.9}Ga_{0.1})_2O_3$ /GaAs substrate after oxidation and additional CAIBE (left). The right portion of each figure represents a 10× enlargement of the etched holes on the left. In order to evaluate the etch depth and profile of our samples,

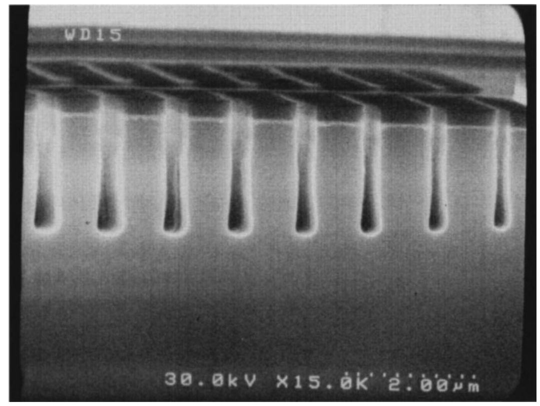


FIG. 4. Cross-section view of eight cleaved lines after oxidation and two additional CAIBE.

we have also fabricated line gratings in addition to hole arrays. The line gratings are etched using the same procedures, and are cleaved prior to electroplating. Figure 4 shows a cross-section view of eight lines that have been cleaved after three CAIBE etching procedures (2 mins each run), with the top darker layer being the remaining $(Al_{0.9}Ga_{0.1})_2O_3$ mask. The average width of the lines increase from 190 nm (right) to 410 nm (left) in the image, and have etch depths that range from 1.8 (right) to 2.1 μm (left). These correspond to aspect ratios of approximately 9:1 to 5:1, respectively. The sidewall profile and etch depth of the trenches in the figure suggest the high anisotropy of the CAIBE process, as well as the robustness of the $(Al_{0.9}Ga_{0.1})_2O_3$ layer as an ion etch mask for the GaAs substrate.

B. Steam oxidation

During the oxidation procedure, the $Al_{0.9}Ga_{0.1}As$ layer underneath the GaAs cap is oxidized laterally through the hole array patterns. The oxidation rate is highly dependent on the Al content in the AlGaAs layers as well as the furnace temperature. Thus, calibration for the specific composition and thickness of the AlGaAs layer, as well as the furnace environment is necessary. Figure 5(a) shows an SEM image of an array of etched holes of 225 nm diameter, 0.5 μm apart in the y direction and 1 μm apart in the x direction, that has been oxidized partially in the lateral direction. The oxide extent is 250 nm after 30 mins in the furnace environment described here. The oxidation process seems to be isotropic in the $Al_{0.9}Ga_{0.1}As$ layer. The unoxidized portion of the AlGaAs layer has been removed by further CAIBE. This is more obvious in the 38° tilted view of the same array in Fig. 5(b), where the darker background is the GaAs substrate, and the brighter stripe-like structures are the $(Al_{0.9}Ga_{0.1})_2O_3$ formed around the hole array patterns. The sidewalls of the etched holes can be observed through the $(Al_{0.9}Ga_{0.1})_2O_3$ layer, and they reveal the high anisotropy of the ion etching process. Although the structures in Fig. 5 are sufficient for electroplating Ni columns, it is preferable to have a very smooth and robust surface, including the areas between the columns, for proper slider contact in subsequent magnetic

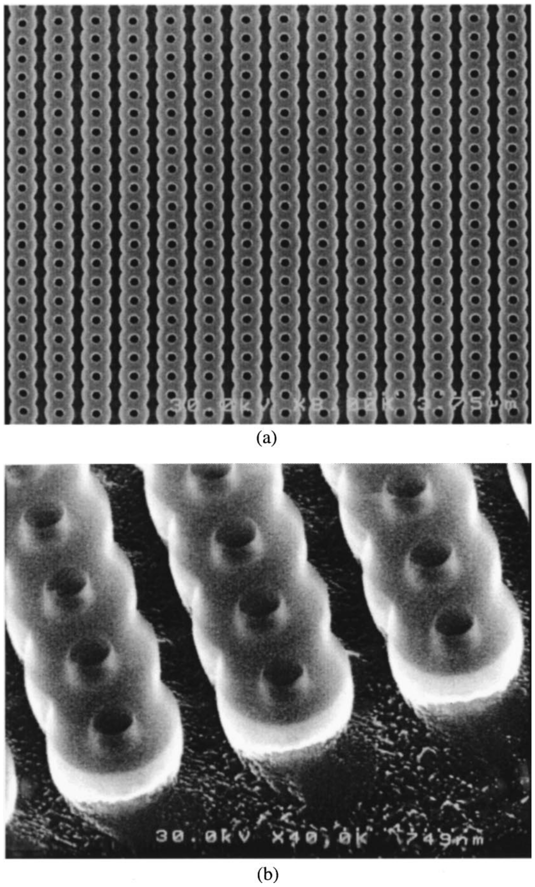


FIG. 5. (a) SEM image of an array of etched holes of 225 nm, 0.5 and 1 μm apart in the y and x direction, respectively, that has been partially oxidized in the lateral direction. (b) A 38° tilted view of the same array in (a).

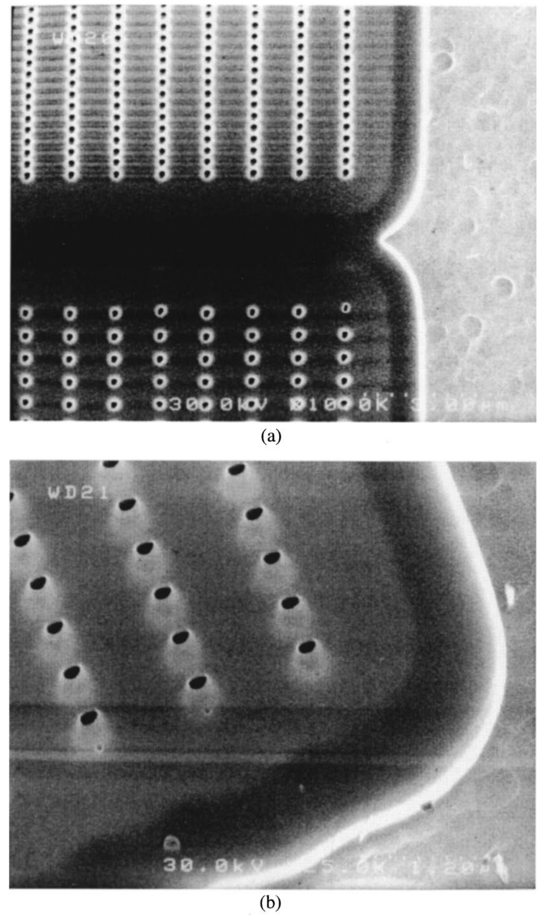


FIG. 6. (a) Top view of two track arrays that have been completely oxidized in between the columns even in the cross-track direction (1 μm spacing). (b) A 38° tilted view of the bottom array (0.5 μm spacing in the down-track direction) in (a).

characterization. Therefore, longer oxidation times are necessary before additional CAIBE and electroplating. Figure 6(a) shows a top view of two track arrays that have been completely oxidized between the columns even in the cross-track direction (1 μm spacing). The $(Al_{0.9}Ga_{0.1})_2O_3$ extent is 1.67 μm after 2 h of oxidation. Once again, the unoxidized AlGaAs has been etched away by CAIBE after oxidation, and the $(Al_{0.9}Ga_{0.1})_2O_3$ mask remains intact, as can be seen in the tilted view in Fig. 6(b).

C. Electroplating into semiconductor

The electroplating of magnetic materials into semiconductor is still not a well-characterized process, and the quality of the electroplated magnetic structures was initially a concern. Our recent results[6] have indicated, however, that the Ni columns plated in etched holes in a conductive GaAs substrate have similar magnetic properties to the Ni columns plated from a metallic seeding layer. We therefore continue to use electroplating to form the Ni columns used in our patterned media work. Figure 7 shows a 50×50 electroplated Ni column array of 200 nm diameter, with a 0.5 μm spacing down track and a 1 μm spacing cross track, embedded in an $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs substrate. Pulse plating is preferred over dc plating since the former is believed to have more ''throwing'' power, which should facilitate the filling of Ni into the high aspect ratio holes in the substrate.[10] It can be seen from Fig. 7 that each hole is evenly filled, and the plating seems to be very uniform over a large area. The adhesion of the $(Al_{0.9}Ga_{0.1})_2O_3$ layer to the semiconductor substrate is critical during the electroplating process. If the adhesion is poor, we find that the plating may propagate laterally between the $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs layers, and Ni disks will be formed along the interface. This situation is undesirable, and has prompted us to use $Al_{0.9}Ga_{0.1}As$ instead of AlAs for the oxide masking layer. It has been found that the oxide formed from AlGaAs alloys tends to be more mechanically stable against delamination along the oxide/semiconductor interfaces after any post-oxidation thermal cycling or processing steps which require temperatures of $>100\,°C$.[11] With the addition of the thin graded layer of AlGaAs between the GaAs and AlGaAs layers, the adhesion of the mask prior to the final etch has also been further enhanced. Another advantage of using AlGaAs alloys is that the linear shrinkage of oxidized AlGaAs layers (formed above 300 °C) is less than that from AlAs layers.[7] The reduced shrinkage is preferable in order to preserve the small diameters of the defined holes.

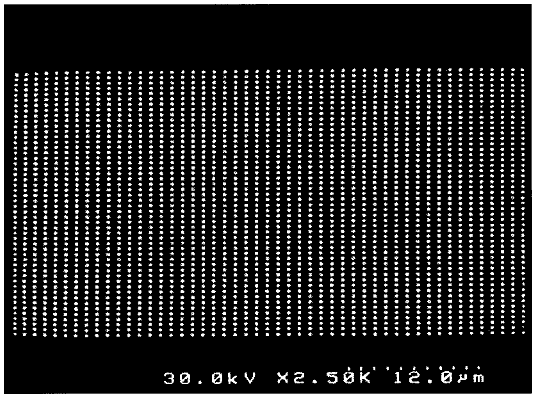


FIG. 7. 50×50 electroplated Ni column array of 200 nm diameter, with a 0.5 μm spacing down track and a 1 μm spacing cross track.

D. Magnetic characterization

Magnetic force microscopy (MFM)[12] is initially used to characterize the magnetic properties of the patterned media fabricated using the procedures described here. In the MFM measurement, we use a home made MFM utilizing electrochemically etched nickel tips[13] and a fiber-optic interferometer[14] as the vibration detector. Figure 8(a) shows a SEM image of an array of Ni columns embedded in an $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs substrate. The columns are 200 nm in diameter, and are spaced 0.5 and 1 μm in the down-track and cross-track direction respectively. Figure 8(b) shows a MFM image of a 4 μm×4 μm scan of the same set of plated Ni columns in (a). The black and white contrast seen in the MFM image represents the two possible magnetization states, which are parallel or antiparallel to the long axis of the cylindrical columns. Figure 9(a) shows a SEM image of a similar array as in Fig. 8(a), except the columns are 120 nm in diameter, and have a 0.25 μm down-track spacing. Figure 9(b) shows a MFM image of an electroplated Ni column array of the same configuration as in (a).

Once the electroplated Ni columns are confirmed to be magnetic using MFM, we characterize the data storage capabilities of our structures using scanning magnetoresistance microscopy (SMRM).[15] In the SMRM technique, a slider (consisting of commercial inductive write poles and a MR or spin valve read sensor) that is used in conventional magnetic recording is placed in contact with the sample. The smoothness of the $(Al_{0.9}Ga_{0.1})_2O_3$ surface and the embedded structure of the Ni columns allow minimum distance between the read sensor and the medium. This is critical for maximizing the resolution and magnitude of the signal from individual magnetic columns. Our previous results have shown that the spatial extent of the column response in the down-track direction is on the order of 500 nm,[6] which should still allow us to resolve each individual column when the down-track separations are 500 nm. Further reduction in the distance between the columns (250 and 125 nm) will enable us to study the interesting regime of patterned media storage, where the signals from adjacent columns are interfering with the signal from the column directly below the read sensor.

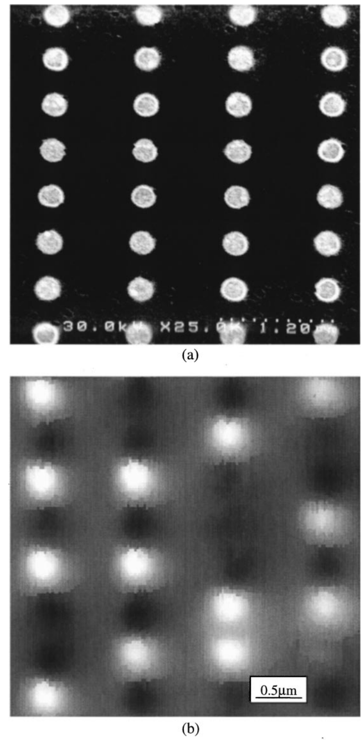


FIG. 8. (a) SEM image of an array of Ni columns embedded in an $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs substrate. The columns are 200 nm in diameter, and spaced 0.5 and 1 μm in the down-track and cross-track direction, respectively. (b) A MFM image of a 4 μm×4 μm scan of the same set of plated Ni columns as in (a).

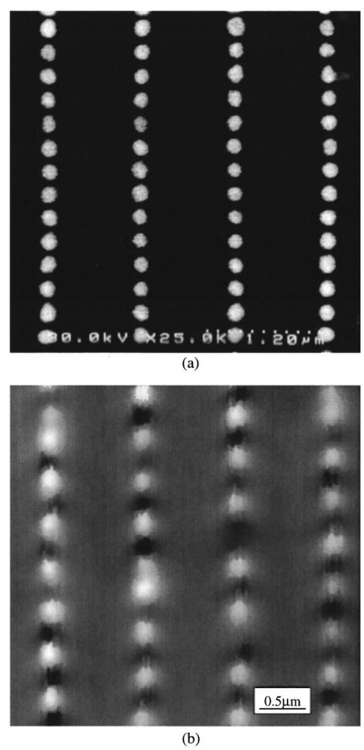
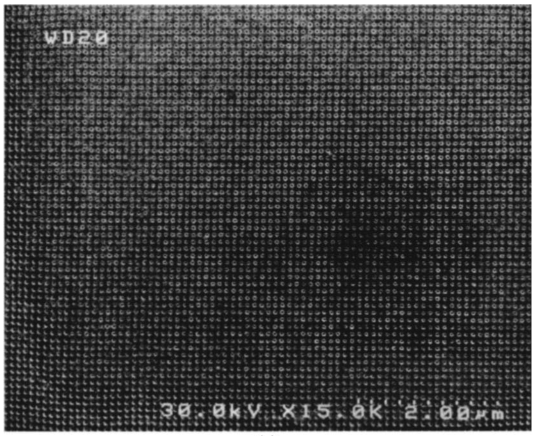
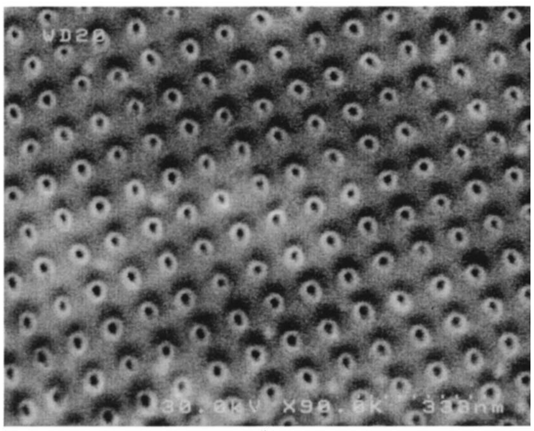


FIG. 9. (a) SEM image of an array of Ni columns embedded in an $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs substrate. The columns are 120 nm in diameter, and spaced 0.25 and 1 μm in the down-track and cross-track direction, respectively. (b) A MFM image of a 4 μm×4 μm scan of an electroplated Ni column array of the same configuration as in (a).



(a)



(b)

FIG. 10. (a) SEM image of a 100×100 array of etched holes of 18 nm diameter and 100 nm spacing in an $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs substrate. (b) A close-up view of the array in (a).

E. High density structures

In addition to the magnetic characterization of the structures shown in this report, work is now in progress to fabricate and characterize higher density patterned media structures. Using electron beam lithography, we have patterned large arrays of holes of 18 nm diameter and 100 nm spacing (65 Gbits/in.^{2}) into a 450-nm-thick PMMA layer, which again served as both the *e*-beam resist and an ion etch mask. We have used Cl_2 assisted ion beam etching to transfer the hole patterns into the $Al_{0.9}Ga_{0.1}As$ layer, which is converted into a robust $(Al_{0.9}Ga_{0.1})_2O_3$ mask during wet thermal oxidation. Figure 10(a) shows a SEM image of a 100×100 array of etched holes of 18 nm diameter and 100 nm spacing in an $Al_{0.9}Ga_{0.1}As$/GaAs substrate. A close-up view of the array is shown in Fig. 10(b). One difficulty with the smaller diameter holes is the slower etching rate compared with the bigger diameter holes. If the first CAIBE stops in the $Al_{0.9}Ga_{0.1}As$ layer instead of the GaAs substrate, the bottom of the hole patterns will be converted to $(Al_{0.9}Ga_{0.1})_2O_3$ after oxidation. When this happens, any further transfer of the hole patterns into the GaAs substrate will be very difficult, and in some cases, impossible without the complete degradation of the $(Al_{0.9}Ga_{0.1})_2O_3$ masking layer. One solution will be to use

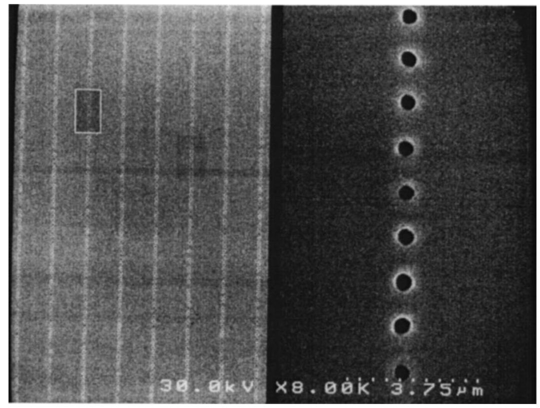


FIG. 11. SEM image of an array of etched holes of 35 nm diameter, spaced 0.125 and 1 μm apart in the *y* and *x* direction, respectively (left). A 10× enlargement of eight of the holes is shown on the right.

thicker PMMA masking layer, provided that the high resolution of the *e*-beam lithography is preserved. $Cr/Au/SiO_2$ masking layers can also be used in conjunction with a thin layer of PMMA.[4] A thinner AlGaAs masking layer may be sufficient as well, since smaller diameter holes will require shallower etch depths for the same aspect ratio.

For data storage demonstration purposes, we are concurrently pursuing denser Ni columns in the form of tracks by further reducing the spacing in the down-track direction, while keeping the 1 μm spacing in the cross-track direction. Figure 11 shows a SEM image of an array of etched holes of 35 nm diameter, spaced 0.125 and 1 μm apart in the *y* and *x* direction respectively, in an $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs substrate. The ability to record in the individual magnetic columns in this 5.2 Gbits/in.^{2} configuration will demonstrate perpendicular patterned media promise for high density data storage. This areal density would be greatly enhanced when narrower read sensors become available and the tracks are spaced closer than 1 μm in the cross-track direction.

IV. CONCLUSION

We have presented in detail the procedures for making perpendicular patterned media structure consisting of cylindrical high aspect ratio single domain Ni particles, embedded in a durable nonmagnetic $(Al_{0.9}Ga_{0.1})_2O_3$/GaAs substrate. The technique involves the use of PMMA as both an *e*-beam sensitive resist and an ion beam etch mask. The high aspect ratio of the Ni columns is achieved through the high anisotropy of the chemically assisted ion beam etch as well as the superb selectivity of GaAs over $(Al_{0.9}Ga_{0.1})_2O_3$ during CAIBE. Electroplating is used to deposit Ni into the holes in the semiconductor substrate. We have fabricated Ni columns in the form of tracks, and have used MFM to characterize the plated Ni. Current work involves further characterization using MFM and SMRM, as well as the fabrication and demonstration of data storage in higher density patterned media structures.

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