MAGNETIC NANOSTRUCTURES FORMATION VIA LOCAL ANODIC OXIDATION AND MAGNETRON SPUTTERING THROUGH LITHOGRAPHIC MASK
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Resume
Changes of magnetic properties of thin metallic films caused by the magnetron sputtering technique and by oxidation lithography were studied. The magnetic nanostructure formation was done via a lithography by sputtering the material through a mask. The anodic oxidation lithography (LAO) via scanning probe microscopy (SPM) device Ntegra Aura was used to attempt to create the magnetic nanostructures. At the beginning, the work has been scoped to the preparation and testing multilayered magnetic nanostructures based on different combinations in material composition of layers. The magnetic properties of these samples were examined by Kerr phenomenon. We observed increasing of the magnetic hardness of the magnetic nanostructure when the copper layer in the composition of ferromagnetic and antiferromagnetic multilayers has been replaced by an aluminium layer. Adding the aluminium layer onto the copper layer resulted in a decrease of magnetic hardness. This work was followed by creating of horizontal nanostructures by LAO. Changes induced by the LAO observed in the magnetic properties of the samples correspond mainly to topographical changes of the surface. Changes in the magnetic properties induced by LAO or by a variability of sputtered layers could lead to the further production of magnetic printed circuit or photonic nanostructures.

1. Introduction
Investigating the nanomaterial properties and new directions of using the acquired knowledge is driving the nanotechnology through time. Multilayers consisting of magnetic and non-magnetic metals were studied since the 90s of the last century. Combination of thin magnetic layers interlaced with thin non-magnetic layers leads to the giant magnetoresistance effect (GMR) commonly used in industry as integrated GMR sensors, such as magnetometers, differential sensors or magnetic field direction sensors (angle GMR), e.g. magnetic sensor for automotive applications [2]. Nowadays GMR elements production consists mainly of a vacuum materials deposition on a silicon substrate and subsequent modification of the area by thermal annealing and magnetic annealing. Using photolithography, the surface is adapted so that the sensor has the greatest resistance and for its measurement small current is sufficient. Resistors of 10 kΩ were already formed as 2 μm serpentine traces covering an area less than 100 μm square [3]. Resistor areas are then involved in the Wheatstone bridge, where a change of the magnetic field is converted to an electrical voltage. Magnetic thin films are generally formed of iron, cobalt, nickel, or their alloys. Non-magnetic thin films are for example
As the temperature rises during the preparation of the copper and cobalt multilayers, the GMR effect decreases due to the mixing of these two materials [4]. Thanks to their magnetic performances, the materials such as cobalt and copper also have a potential use in magneto-optics, especially in the construction of photonic crystals [5–8]. Reaching the dimensional limits of formation of nanostructures based on the magnetic thin films is what connects GMR electronics and photonic crystals.

In our work we present possibilities for creating structured surfaces, whether applying a metallic material by magnetron sputtering through a mask, or a combination of planar sputtered sample with subsequent surface modification by applications of local anodic oxidation (LAO). The LAO is enabled by applying a bias voltage between the sample and the atomic force microscopy (AFM) probe that allows to create structures with a width of several tens of nanometers [9, 10].

2. Methodology

Lithographic methods can be divided into two main groups. Top-down methods [11] (material removal layer after layer to achieve the desired structure) and bottom-up methods [11] (material layering, self-organization, etc.). In our study, we focused on two bottom-up methods of transferring a structure on a substrate. They have both a common basis in the magnetron sputtering allowing thin layers deposition from units to hundreds of nanometers under a reduced pressure. In case of the first method, the smallest size of the lithographic mask structures for which this structure is transferred on a substrate at the desired topographical layout by sputtering metal materials was investigated. In case of the other method, the combination of materials and suitable setting of an oxidation lithography was investigated for result into observable change of the magnetic properties of the substrate caused by the oxidation lithography.

2.1 Selection of the materials for magnetron sputtering

The selected materials - cobalt and copper - are typically used in a combination of layers for GMR reading heads of hard disks drives [1]. The principle of GMR is a reduced possibility of an electron to pass through a conductive material. Electron with spin oriented identically with magnetic domains can pass through unlike the electron with the opposite spin, which is reduced. For an antiferromagnetic configuration of the magnetic domains in the cobalt layers the resistivity is higher than for a ferromagnetic arrangement. The ferromagnetic or antiferromagnetic orientation of the individual magnetic layers of the composite material depends on the thickness of the non-magnetic interlayer as well as the rate of the magnetoresistance [4]. The GMR effect increase can be achieved by increasing the number of pairs of layers Co/Cu, adding buffer layers of Fe, or adding Al as interlayer [1]. Therefore we prepared a variation of multilayers Si/Fe/Co/[CuCo]5/Fe, Si/Fe/Co/[CuCo]/Al/[CuCo]/Fe, Si/Fe/Co/[CuCo]/Al/Co/[CuCo]/Fe and Si/Fe/Co/[CuCo]/Al/[CuCo]/Fe, where the Cu/Co interlayers thickness was chosen with respect to the antiferromagnetic ordering of the magnetic cobalt domains, as stated Parkin [4].

2.2 The oxidation rate analysis of the selected materials

Both nanostructure creation methods require sputtering various materials and therefore it is necessary to swap the sputtering targets within a single procedure. Each material target change, however, leads to sample chamber opening and exposing the already sputtered material to the influence of the ambient (oxidizing) environment. A native
oxide layer forms on each material and the composite material thus becomes poorly definable. The following Table 1 indicates the tendency of our chosen materials to react with oxygen. Oxides listed in Table 1 are the most common types formed by oxidation at room temperature. The column "E" refers to the energy that must be supplied to the system, to allow spontaneous oxidation. If the oxidation energy is positive, the material is stable. In the case of negative energy the spontaneous oxidation takes place. The more negative the value of this energy, the faster the material should oxidize. However the practical measurements as described in [12 – 14] show that the oxidation process is more complex, and for some materials the assumed oxidation rate does not correspond with actual measured values (Table 1, column "P"). The oxidation rate values for aluminum [12], iron and copper [13] and the cobalt [14] are related to the ambient temperature and normal levels of the relative humidity, i.e. 30 % - 50 %.

For the relative humidity values between 60 % and 70 %, or by increasing the ambient temperature, the aggressiveness of the environment increases and the resistivity of the material decreases so that the oxidation takes place more easily. The progress of the oxidation rate has a logarithmic course, when after the first minute of the material exposure only a slight growth of oxide layer occurs [12].

### 2.3 Magnetron sputtering

The principle of the magnetron sputtering lies in a bombardment of a solid material target by energetic particles (ions of an inert gas), thereby the material from the target is released and deposited on the substrate. The entire process takes place under a reduced pressure. The process gas (argon) is introduced into the chamber, where in the front of the material target a glow discharge is initiated by applying a voltage. The sputtering is suitable for a deposition of conductive material layers, which can thus be prepared more effectively in a comparison with chemical processes. The quality of a deposited layer is affected, besides the material target purity, by the biased voltage and by the rotation speed of the substrate table [15].

### 2.4 Formation of nanostructures using scanning probe microscopy

The process of an intentional deformation of the surface of the sample is known as lithography and can be realized either by a mechanical or an electrochemical force. The mechanical force lithography in our case was used only as a tool for a division of the sample into smaller segments, on which an electric (chemical) lithography – the local anodic oxidation (LAO) was implemented. For the LAO the only requirement for the environment is a high relative humidity in the nearest area of the sample. The entire system must be closed in order to control the degree of the relative humidity.

### Table 1

<table>
<thead>
<tr>
<th>Material</th>
<th>Oxide</th>
<th>E (kJ mol(^{-1}) oxygen)</th>
<th>P (nm/30 s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>Al(_2)O(_3)</td>
<td>-1045</td>
<td>0.6</td>
</tr>
<tr>
<td>Fe</td>
<td>Fe(_2)O(_4)</td>
<td>-508</td>
<td>2.0</td>
</tr>
<tr>
<td>Co</td>
<td>Co((\text{OH}))(_2)</td>
<td>-422</td>
<td>0.8</td>
</tr>
<tr>
<td>Cu</td>
<td>CuO</td>
<td>-254</td>
<td>1.0</td>
</tr>
</tbody>
</table>

The relative humidity has the most significant effect on the course of oxidation. Below 60% of the relative humidity the oxidation progress is moderately strong and only the first few nanometers of the material oxidize. The thickness of the native oxide layer is stable after about the first minute of the exposure to the material oxidizing influence and further increases slightly. This layer forms a sort of barrier between the pure material and oxidizing environment.
In the environment with the high relative humidity a mechanical contact of submicron areas leads to spontaneous formation of a water meniscus between the tip and the sample. The meniscus is a source of oxyanions necessary for the oxidation of the sample surface. The oxidation is initiated by applying a voltage “U” to the tip [16]. The size of oxidation traces is primarily affected by the sharpness of used tip, but also by other parameters, for example by the value of bias voltage, the time for which the bias voltage is applied and by the inclination of the material to oxidize. The height of the trace and its continuity is indirectly dependent on the rate of a shift of the tip during the process.

### 3. Material and experimental methods

The used materials were prepared from material targets supplied by Quorum Technologies, namely: TK8900 Cobalt (Co) 0.3 mm thick; TK8897 Iron (Fe) 0.1 mm thick; TK8875 Aluminium (Al) 1 mm thick; SC502-314H Copper (Cu) 0.1 mm thick.

The multilayered films were sputtered on laboratory glass slides Thermo Scientific – Menzel-Gläser, extra-white soda-lime glass. The slide glass was treated in an ultrasonic bath: firstly in a detergent and then washed in distilled water, followed by an ultrasonic bath in acetone and methanol, both for 10 minutes. The slides were dried by compressed nitrogen and for the case of the direct sputtering were put into the deposition chamber of the coating machine Q150T ES located at laboratories of the Palacky University in Olomouc. The vacuum chamber was filled with argon at the operating pressure range between $5 \times 10^{-3}$ and $5 \times 10^{-1}$ Pa. When combining multiple materials per sample it was always necessary to open the chamber and swap the targets. The target change time was minimized to 30 seconds. During this time the top 2 nm of iron oxidize. For cobalt and copper it was about 0.8 - 1 nm in this time period (see Table 1). During the sputtering process the layer thickness was taken into account so the sputtering/operating current was set to 25 mA for sputtering copper, 30 mA for cobalt and 50 mA for iron and aluminium. The layer deposition was carried out according to the Table 2.

<table>
<thead>
<tr>
<th>Layer no.</th>
<th>Material</th>
<th>I (nm)</th>
<th>II (nm)</th>
<th>III (nm)</th>
<th>Mask IV (nm)</th>
<th>Mask V (nm)</th>
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<tbody>
<tr>
<td>1</td>
<td>Fe</td>
<td>5.0</td>
<td>5.0</td>
<td>5.0</td>
<td>5.0</td>
<td>5.0</td>
</tr>
<tr>
<td>2</td>
<td>Co</td>
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<td>1.2</td>
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<td>1.2</td>
<td>1.2</td>
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<tr>
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<td>1.1</td>
<td>1.1</td>
</tr>
<tr>
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<td>-</td>
<td>-</td>
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<tr>
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<tr>
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<tr>
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<td>1.1</td>
<td>1.1</td>
<td>1.0</td>
<td>1.1</td>
</tr>
<tr>
<td>12</td>
<td>Cu</td>
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<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>13</td>
<td>Co</td>
<td>1.1</td>
<td>1.1</td>
<td>1.1</td>
<td>1.0</td>
<td>1.1</td>
</tr>
<tr>
<td>14</td>
<td>Fe</td>
<td>5.0</td>
<td>5.0</td>
<td>5.0</td>
<td>20</td>
<td>5.0</td>
</tr>
<tr>
<td>Sum (nm)</td>
<td>-</td>
<td>26.8</td>
<td>32.4</td>
<td>30.4</td>
<td>40.9</td>
<td>26.7</td>
</tr>
</tbody>
</table>

Table 2

The composition of the tested multilayered films is shown in columns I, II, III, mask IV and mask V. In each row there is the layer number, chosen material and layer thickness. The layers are numbered starting from the silicon substrate. The absence of a layer in the composition is marked with a “-“.
3.1 Creating structures using a mask

The combination of layers in Table 2 labeled as "mask IV" and "mask V" were directly sputtered on the substrate through a mask. As a mask the copper grid below the samples for TEM was used, namely G200 Gilder Grids, 200 mesh and Quantifoil R 3,5/1 Cu-200QC3/1, Cu grid, 200 mesh with an additional membrane 5 mesh. After the detergent, the alcohol and the nitrogen treatment the mask was fixed on the glass slides so that the surface adheres well to the treated substrate. The substrate prepared this way was placed in a chamber of the coating machine and the process of sputtering a material according to the conditions above and in the order of that in Table 1 columns "mask IV" and "mask V" started. The mask was stripped off after sputtering and the sample was ready to characterize.

3.2 Lithography by local anodic oxidation

The lithography by local anodic oxidation was performed using the scanning probe microscope Ntegra Aura in the contact mode of the atomic force microscopy, in the vector mode of oxidation lithography respectively, using the probe DCP20 supplied by the NT-MDT company. The oxidation was carried out for a different time duration from 500 ms to 2000 ms and for a different applied voltage from -2 V to -8 V at an artificially increased relative humidity in the range 65 – 78 %. For the purposes of the local anodic oxidation simple combinations of materials were layered. A bilayer of 20 nm Fe/ 5 nm Co was deposited on the glass slide, where cobalt forms a layer mitigating the environmental oxidizing influences so that the Fe layer is protected and therefore capable of the controlled oxidation. On the multi-layer material 5 nm Co/ 5 nm Fe/ 5 nm Co a change of the magnetic response caused by the oxidation lithography was measured. For a magnetic measurements by the magnetic force microscopy a probe NSG01/Co was used.

4. Results

Changes in the magnetic response caused by a change in the composition of the sample layers were shown by a measurement of the hysteresis loop using the Kerr phenomenon. The scanning electron microscope (SEM) VEGA 3 LMU by Tescan company and the scanning probe microscope Ntegra Aura by the NT-MDT company in the mode of atomic force microscopy (AFM) were used to characterize the morphological and topographic properties of the prepared samples. The characterization of the magnetic properties of the samples was performed by a magnetic gradient measurement using the magnetic force microscopy (MFM) in the dual scanning mode where the topography of the sample is measured by a semi contact AFM. The information about magnetic force sensed between the probe and the sample is collected by rescanning the already measured line of the topography of the sample using the phase shift or the frequency of the tip in a certain predetermined height above the sample surface.

The normalized magnetization of the composite material - sample I on an external magnetic field is shown in Fig. 1. The step on the hysteresis curve originates from different coercivities of cobalt and iron and is observable only for a specific angle of the sample toward the applied magnetic field. The magneto optical response of samples I, II and III measured for the same applied magnetic field is shown in Fig. 2. Inserting the aluminum layer, respectively aluminum oxide into the composition leads to a noticeable decreasing of the coercive field and a remarkable shift of the hysteresis curve towards the positive pole of the external magnetic field. The hysteresis loop of the sample III compared to that of the sample I, however, shows an increase of the coercive field, which is caused by replacing a thin layer of copper by a twice as thick layer of aluminium, aluminium oxide respectively.
Fig. 1. The sample I - Fe/[Co/Cu]5/Cu/Fe: the hysteresis curve of magnetization on external magnetic field, where $M$ is the total magnetization of the sample and $M_s$ is the saturation magnetization. A step marked by the grey circle has an origin in different coercivities of the cobalt and iron.

Fig. 2. The samples I, II and III: Hysteresis loops of the Kerr effect for samples I - Fe/[Co/Cu]5/Cu/Fe (black), II - Fe/[Co/Cu]2/Al/[Co/Cu]3/Fe (solid grey) and III - Fe/[Co/Cu]2/Al/[Co/Cu]2/Fe (dashed grey).

Fig. 3. The homogeneity characterization of the sputtering process on the sample II - Fe/[Co/Cu]2/Al/[Co/Cu]2/Fe: the black curve characterizes the detection point of the sample closer to the center of the sputtering sample holder, the grey curve characterizes the same sample but the detection point of the sample was located at the edges of the sputtering stage.
In Fig. 3 two hysteresis curves are plotted, both for the sample II. The difference between these two curves has an origin in an unbalanced sputtering process - the values of the thicknesses of the individual layers for the gray curve are smaller and the layers are likely to be inhomogeneous.

After the characterization of the surface magnetic properties, the next phase of the research took place. The material composition of the sample I has been sputtered through a lithographic mask as described above.

In the left hand side of the Fig. 4 the sputtered multilayer "mask IV" through a mask G200 is shown. The dimensions of the structure measured by the SEM and in parentheses dimensions of the masks provided by a supplier are as follows: Pitch 120 μm (125 μm), Hole width 92 μm (90 μm), Bar width 34 μm (35 μm). From the image and measurement it is obvious that the sputtered structure does not have a good integrity - the region collapsed. The sample "mask IV" sputtered through a mask Quantifoil R 3.5 / 1 is shown in Fig. 4 on the right. During the SEM measurements some Quantifoil membrane fragments were observed which means the membrane collapsed during the sputtering process.

From the atomic force microscopy measurement (Fig. 5) on the left the height difference of a sputtered square structure and the area covered by the mask was determined as 40 nm, which corresponds to the sum of amount of the material sputtered in the individual layers according to Table 2. column "mask IV" (40.9 nm). The height of the spherical structure of the sample on the right varies from 2 to 4 nm instead of the expected 26.7 nm, from which we conclude that the collapse of the membrane of the masks occurred soon after the start of the sputtering.

The AFM measurement image of LAO performed on the 20 nm thick iron layer with a 5 nm cobalt cover is shown in Fig. 6. The oxidation was performed for different applied voltages (columns) and for various time exposures of the voltage at one point of the curve (lines). Although the oxidation trace for the lowest applied voltage setting and the shortest oxidation time is apparent, in case of the largest negative voltage setting applied for the longest time the oxidation did not take place. Same for some medium voltages and times. The height of an oxidation trace within one single structure ranges from a few nanometers to several tens of nanometers (e.g. for a circle oxidized at voltage of -8 V and time of 1000 ms the height of the trace ranged from 3.05 to 70.8 nm).

Fig. 7 shows a topographic measurements of the substrate 5 nm Co/ 5 nm Fe/ 5 nm Co with an area of an oxidized surface (top left) and the cross 68th line (top right). The MFM image and the cross-section of the same line is shown on the bottom of the Fig. 7.
Fig. 5. The AFM image of structures sputtered through a mask G200 (left - "mask IV") and Quantifoil R 3.5 / 1 (right - "mask V").

Fig. 6. The result of the LAO on 20 nm Fe/ 5 nm Co: circular structures in columns were oxidized at voltages of -2 V, -4 V, -6 V, -8 V from left, for the lines oxidizing time was 500 ms, 1000 ms, 1500 ms a 2000 ms from bottom. The centre of every circular structure lithography is marked with the cross.

Fig. 7. The AFM LAO (upper left), and the MFM (lower left) of 5 nm Co/ 5 nm Fe/ 5 nm Co and the cross section and the same 68th line of the topography measurement (right).
5. Discussion

According to [1], the insertion of an aluminium layer into the composition of the ferromagnetic and antiferromagnetic multilayers has the effect of increasing the magnetorezistivity of the multilayer. It is shown that the substitution of one interlayer of electrically conductive copper for the aluminium (aluminium oxide respectively) leads to an increasing of the magnetic hardness of the material (Fig. 2).

Differently an addition of the aluminium oxide layer (aluminium respectively) to the copper interlayer has led to a shift of the hysteresis curve towards the positive pole of the external magnetic field (Fig. 2). That means only, a minor change of the external magnetic field is necessary for all the domains of cobalt to orient in a new direction of the magnetization. The magnetic domains in two thin adjacent layers arranges ferromagnetically or antiferromagnetically during the sputtering process. The type of the arrangement depends on the thickness of the non-magnetic interlayer [4]. Assuming the thickness of copper layers was chosen considering the antiferromagnetic arrangement, adding the aluminium layer right to an already sputtered copper layer may have resulted in the ferromagnetic ordering of the cobalt domains in the following layer, which would explain the shift of the hysteresis loop.

The homogeneity of the sputtered layers is strongly dependent on the position of the substrate within the sputtering chamber. This dependence is confirmed by the results of the measurement of the hysteresis loops for the prepared sample investigated both in the area that was closer to the center of the substrate holder during the sputtering process and the area on its edge (Fig. 3).

The result of the sputtering lithography is that the polymeric and thin membranes are unsuitable for the sputtering of thin metal layers. The sputtering process led to the collapse of the thin type Quantifoil membrane.

The reason could be in the load on the membrane produced by the sputtered material, or even in a pressure change in the chamber, which is a part of the material target replacement or in a combination of both factors. The copper grid is preferable for this technique but its disadvantage is the size of the structures that can be sputtered. The metal masks with a sufficient resolution are not easily available.

The increasing height of the oxidation traces with a rise of the oxidation time and with an increase of the applied negative voltage fit the findings on a silicon oxidation [16]. The missing oxidative traces on the lithography for this work and the large elevation changes within one oxidizing trace may correspond to an asymmetry in the sputtered cobalt coating layer, so that only in some areas the oxidation of the iron was possible.

6. Conclusions

The multilayered nanostructures Fe/Co/[Cu/Co]/Fe, Fe/Co/[Cu/Co]/Al/[Cu/Co]/Fe, Fe/Co/[Cu/Co]/Al/[Cu/Co]/Fe, Fe/Co and Co/Fe/Co were prepared by the magnetron sputtering method. The first results show possibilities and certain reserves of the lithographic methods same as reserves in the preparation of the samples. The used sputtering device has only one material target holder so during the material target change the already sputtered layer was exposed to the influence of the ambient (oxidizing) environment. The oxidation of the sputtered layer was minimized only by shortening the time of change of the material target, so these layers are not well material defined. Even so these multilayers are sufficient for a preliminary study of the lithographic methods.

The basic magnetic characterization of the prepared multilayers Fe/Co/[Cu/Co]/Fe, Fe/Co/[Cu/Co]/Al/[Cu/Co]/Fe and Fe/Co/[Cu/Co]/Al/[Cu/Co]/Fe was performed by their hysteresis loops measuring using the Kerr
phenomenon. Adding the aluminium layer into the composite of iron, cobalt and copper, the significant shift of the hysteresis curve was noticed. The rearrangement of the magnetic domains of the 4th cobalt layer from the antiferromagnetic to ferromagnetic arrangement towards the 3rd layer can be the cause of the curve shift. Substitution of the intermediate copper layer by aluminium has led to an increase of the coercive field of the composed material without a shift of the hysteresis loop and therefore the antiferromagnetic arrangement of the individual layers of cobalt is unchanged. The measurements also confirmed the dependence of the magnetic properties of the sputtered layers on the location of the substrate in the sputtering chamber during the sputtering process.

The formation of nanostructures by sputtering the material through a mask has been successful using the grids below TEM samples. Two types of the grid were used. The lateral dimensions of the sputtered structure through the grid G200 were 125x125 μm. The structures did not have any sharp edges. Probably while removing the mask part of the sputtered material slid off the mask. Sputtering of the structures through the secondary mask Quantifoil R 3,5/1 showed that the structures with lateral dimensions of 4 μm are possible to prepare. However the mask itself was unsuitable due to the membrane material which collapsed after sputtering of the first 5 nm of iron.

On the prepared multilayers Fe/Co and Co/Fe/Co the local anodic oxidation was performed. The topographic deformations by the atomic force microscopy and the rate of magnetic change by the magnetic force microscopy were investigated. The topography of the oxidation trace is strongly dependent on the homogeneity and the thickness of the cover cobalt layer. According to the magnetic response measurement some magnetic changes could have been induced via the local anodic oxidation. However the magnetic response corresponds to the topographical changes more likely.

Both multilayer preparation methods led to the formation of a structured surface. Such a structure of several square mm depending on the size of the mask can be performed by the magnetron sputtering during one single processing. The details of the structures achieve dimensions from tens to several units of μm. For achieving a similar result using the oxidation lithography, a macroscopic translation of the sample is needed. Combining of the individual lithographically modified areas would require a high accuracy of the translation stage. The oxidation trace itself can be even a few hundreds of nm wide which allows an incomparably better resolution of the structure than sputtering through a metal mask enables.

For an optional application of the lithography by sputtering and by oxidation for example in the field of the photonic crystals, the resolution and the material composition of the mask is necessary to solve as well as the collapse of the structure after removing the mask. One possible solution could be a combination with the method of ion beam lithography. An essential step is the use of a sputtering apparatus with more material targets so that during the sputtering process contamination of the substrate is eliminated. In the case of the local anodic oxidation the first step of improvement a change in preparation of the sample is crucial. That means to find out such configuration for which the covering cobalt material is distributed homogeneously.

Acknowledgements
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