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# Fabrication of High-Resolution Gamma-Ray MMCs with AgEr Sensor and Thick Electroplated Absorbers

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**Abstract** We are developing metallic magnetic calorimeters (MMCs) for high resolution gamma-ray spectroscopy for non-destructive assay of nuclear materials. Absorbers for these higher-energy photons can require substantial thickness to achieve adequate stopping power. We developed a new absorber fabrication process using dry film photoresists to electroform cantilevered, thick absorbers. Gamma detectors with these absorbers have an energy resolution of 36 eV FWHM at 60 keV. In this report we summarize modifications to STAR Cryo's Delta 1000 process for our devices and describe the new absorber fabrication process.

**Keywords** MMC • metallic magnetic calorimeter • gamma ray spectroscopy • non-destructive assay • thick absorber • dry film photoresist

## 1 Introduction

High resolution gamma-ray spectroscopy using Metallic Magnetic Calorimeters (MMCs) has already demonstrated improved accuracy over high-purity germanium (HPGe) detectors for non-destructive assay (NDA) of nuclear materials [1].

MMCs are low temperature particle detectors which measure energy of the absorbed particle by measuring a magnetization change [2]. It has an absorber that is thermally coupled to a paramagnetic sensor whose magnetization can be measured using a SQUID magnetometer. For the gamma-rays the absorbers need to be up to a few hundred microns thick to have enough stopping power [3, 4].

Our group has been developing gamma-ray MMCs that integrate the SQUIDs and sensing coils to increase flux coupling and signal to noise ratio [5, 6]. Fabrication of the absorbers for such MMCs is challenging because SQUIDs are sensitive to chemical attack and elevated processing temperatures. We have recently reported a complete fabrication process to electroform cantilevered Au absorbers by using AZ125nXT photoresist to define the tops and a sacrificial, sputtered Cu blanket layer to define the posts [5]. Although this process was successfully applied on several test structures, on the real MMC chip we found that film stress of the sputtered Cu damaged underlying structures. We found that the Cu film stress problem could be eliminated if the film was evaporated rather than sputtered. However, an easier fabrication process was highly desirable to reduce the time and frequent rework required for the Cu/AZ125nXT process.

In this report, we introduce a new thick absorber fabrication process by using two dry film photoresists to define both posts and tops. The new process is completely compatible with the STARCryo Delta 1000 SQUID microfabrication process, enabling future commercial deployment of our integrated SQUID/sensor detector designs. Using this approach, we have completed fabrication of 14-pixel arrays of integrated SQUID/sensor MMCs with attached absorbers.

## 2 Fabrication

The MMCs used in this paper were designed by UNM and fabricated in collaboration with STARCryo. Description of all layers is given in Table 1. Steps 1-8 are performed on the full 4" wafer and are a modified version of the commercial STARCryo "Delta 1000" SQUID process. These steps define the trilayer [7] and two Nb wiring layers from which the SQUIDs, sensing coils and circuits, and magnetizing circuits are made. The wafer is then diced into cm-scale MMC chips and layers 9-13, the Ag:Er paramagnetic sensor, gold thermalization layer, Nb superconducting cap and cantilevered gold absorbers are added.

**Table 1** MMC Process Summary.

| # | Layer | Description | Thickness (nm) |
|---|---|---|---|
| 1 | Nb/Al-$AlO_x$/Nb | JJ Definition & Wiring | 400 |
| 2 | Nb:Ta62 | Passive Persistence Shunts | 220 |
| 3 | $SiO_2$ | Insulation | 130 |
| 4 | AuPd | SQUID Shunt Resistor | 170 |
| 5 | Nb | Second Wiring | 480 |
| 6 | $SiO_2$ | Insulation | 150 |
| 7 | Nb | Third Wiring | 480 |
| 8 | Au | Pads | 330 |
| 9 | Ag:Er | Paramagnet | 1500 |
| 10 | Au | Thermalization | 220 |
| 11 | $SiO_2$ | Insulation | 200 |
| 12 | Nb | Superconducting Cap | 370 |
| 13 | Au | Absorber (Posts + Top) | 20k + 30k |

2.1 Modifications to ”Delta 1000” Process for MMC

The Delta 1000 process was modified to increase the thickness of all wiring layers up to about 500 nm. A second modification to the Delta 1000 process was to reduce the thickness of the $SiO_2$ insulation layers, primarily to increase magnetic coupling of the MMC sensing coils to the paramagnet, but measurements with test structures indicated that this also improved the current-carrying capacity of the vias. For our current MMC wafer, the target oxide layer thickness was 150 nm. Although similar thicknesses worked well on test structures, we found random electrical shorts on the wafer. We have recently found that a subtle problem with the PECVD $SiO_2$ tool may have been responsible for these shorts. That problem has been fixed and we plan to test the thin oxide again. The third change to the Delta 1000 process was to add a step do deposit Nb:Ta alloy passive persistent-current switches [6, 8] onto trilayer wiring.

2.2 Cm-scale Chip Level Fabrication

First step of the cm-scale chip level process is deposition of the paramagnetic sensor material. To address parasitic heat capacity problem in Au:Er sensors due to quadrupole splitting of Au nuclei in the presence of Er ions [9] we are exploring the use of Ag as a host material. 1000 ppm Ag:Er paramagnet is synthesized in homemade induction furnace and sputter deposited on to the chip in dedicated sputtering system [10].

Then Au thermalization layer is electroplated over the paramagnetic sensor and it is followed by PEVCD $SiO_2$ blanket insulation layer to isolate the paramagnet from following superconducting Nb layer. Next Nb superconducting cap layer is sputter deposited to increase magnetic field concentration in the paramagnet [11]. There are circular openings patterned in the superconducting cap that provide access to the paramagnet underneath. As a last step cantilevered Au absorbers are electroplated.

2.3 Novel Cantilevered Absorber Fabrication Process

Two layers of MX5000 series dry-film photoresists from DuPont is used to define molds for cantilevered absorbers. 20 $\mu$m thick MX5020 is used to define posts and 50 $\mu$m thick MX5050 is used the tops. Processing both photoresist layers is very similar with only difference of operation parameters such as exposure and development times. Basic process includes lamination, exposure and development. Prior to lamination of the film the cm-scale chip is cleaned by rinsing with acetone and IPA and then descumming for 5 min under $O_2$ plasma. To laminate the film, the protective polyethylene layer is removed from one side. The film is rolled on sample on hot plate at 90-100 ºC, with gentle pressure applied by a homemade hand roller. Then the photoresist is exposed with 365 nm UV light with intensity of 18 mW/cm$^2$ for 2 sec. Although 20 mW/cm$^2$ or higher intensities are recommended for high resolution, 18 mW/cm$^2$ was maximum intensity available for us. The photoresist is developed in 0.75 wt% $K_2CO_3$ solution at 30 ºC for 1 min (3 min for MX5050). Then the sample is rinsed in DI water and dried with $N_2$ gun. After each of the lamination, exposure and development steps the chip is baked on a 90-100 ºC hot plate for 45 sec. These are applied to enhance adhesion, resolution and resistance to chemicals, respectively.

After posts of the absorbers are defined over the circular holes in Nb cap layer, the $SiO_2$ layer under the table legs is removed by using dry etch to make direct contact to the thermalization layer over the

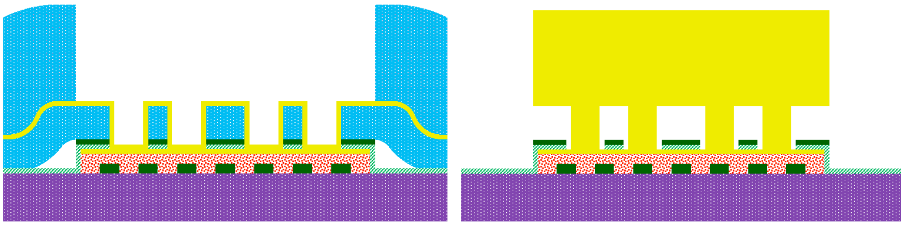

**Fig. 2** *Left,* schematic cross-section of MMC coil area with two layer dry-film photoresist mold ready for plating. Nb cap and oxide layer is opened for the electroformed gold to make direct contact to the paramagnet's gold thermalization layer. *Right,* diagram of same area after the absorber is electroformed and mold is removed (Color figure online).

PM. Then sample is ashed for 5 min with $O_2$ plasma and 250 nm Au seed layer is sputter deposited. Then 50 $\mu$m thick photoresist film is processed to define the tops. Diagram of two-layer mold is shown in Fig. 2 (*Left*).

Electroplating is done is 1 L beaker with 250 ml fresh Technic D7990 RTU solution with Au density of 8.2 gr/L. Vigorous agitation is sustained by using water pump directed toward the chip. 0.25 asd rectangular pulses are applied during plating. After plating both photoresist layers are removed by sonicating in DMSO at 50-60 ºC, typically for 30 minutes. Diagram of the cantilevered absorber after the mold removed is shown in Fig. 2 (*Right*).

SEM images of a completed MMC array is shown in Fig. 3 (*Left*) and on the (*Right*) we see single 475x475x30 $\mu m^3$ absorber on eight 50 $\mu$m diameter posts. The thickness of the absorbers is uniform across the cm-scale chip and as well as single absorber. They are robust, surviving multiple rapid immersions into liquid nitrogen and long sonications in solvents. RRR of meander samples which was electroplated in the same setup is measured to be ~7**.** This is significantly smaller than what we have previously reported with samples made by using Techni Gold 25ES solution [5]. Other plating parameters, such as plating temperature and agitation may have influence on this as well [12].

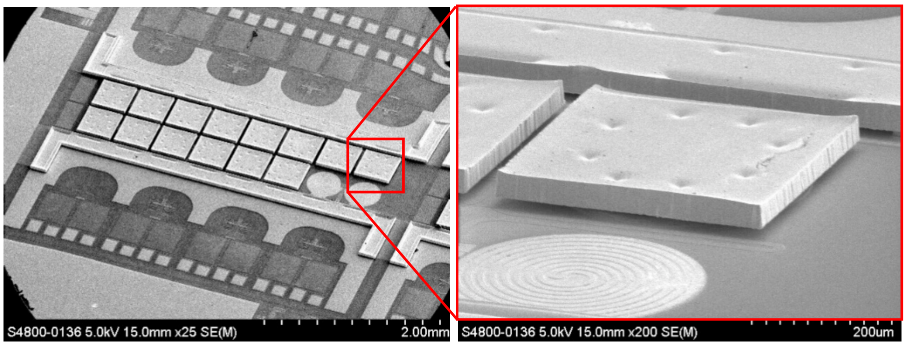


**Fig. 3** *Left* SEM image of a complete 14-pixel MMC. *Right* SEM image of a single 475x475x30 $\mu m^3$ cantilevered absorber on 8 posts of 50 $\mu$m diameter.

Moreover, we have successfully produced 100 $\mu$m thick molds by layering two 50 $\mu$m thick dry film photoresists and exposing them together. Resolution of the molds is slightly degraded, this can be improved by increasing the intensity of the exposure. Additionally, surface finish of electroformed 100 $\mu$m absorbers are not as smooth as 30 $\mu$m absorbers but RRR of a same thickness meander is close to RRR measured on a 5 $\mu$m thick meander which was electroformed in the same setup but with uniform surface finish. This indicates that the surface finish should not degrade any thermalization related performance, which agrees with the experience of the Heidelberg group [13].

The complete devices are characterized at 10 mK in a cryostat with dilution refrigerator at LLNL [6]. Signal rise times of 7 $\mu$s indicate good thermal coupling between the Au absorber and the Ag:Er sensor. Gamma detectors with these absorbers have an energy resolution of 36 eV FWHM at 60 keV and a Gaussian response function. These properties make our gamma-ray MMCs well-suited for high accuracy non-destructive assay of radioactive materials and other applications in nuclear safeguards.

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