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Nanostructured Metamaterials for the thermal and electromagnetic isolation of Cryostats

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ABSTRACT

In terms of thermal and electromagnetic isolation properties, Nanostructured Metamaterials may present important advantages compared to current employed materials. In this work, we present the synthesis routes and the preliminary characterization results of some metamaterials based on Poly-ether-ether-ketone and nanostructured anodic oxides, with low thermal conductivity, combined with Au nanocolumns and three-dimensional nanonetworks. The results reveals proper magnetic and electromagnetic properties and suggest their suitable use under vacuum and cryogenic conditions.

Keywords: Nanostructured Metamaterials, Cryostats, EMC, Thermal Control

1. INTRODUCTION

Thermal and electromagnetic isolation (from DC to GHz range) in ground and space-based cryostats is of utmost importance to ensure the proper working environment of next-generation instrumentation. In the case of X-ray detectors, simultaneous isolation becomes critical for transition edge sensors such as those on the X-IFU instrument (ATHENA mission)[1]. The structural and inner cladding materials of the cryostats are the key to achieving the most demanding insulation levels. In fact, the choice of materials is a delicate balance in which multiple and often contradictory parameters must be balanced, e.g. Weight, vacuum compatibility, electromagnetic reflectance/absorption/transmission and thermal conductivity. However, some engineered nanostructured metamaterials[2, 3] may be appropriate and offer tailored responses and better performances than single-phase/component materials. However, these metamaterials must be compatible with harsh conditions of ultrahigh vacuum and cryogenic temperatures. Furthermore, they should be synthesized through affordable economic routes to be applicable at macroscopic scale. In addition, these materials should be compatible with mature fabrication techniques or new additive manufacturing approaches. In order to achieve these challenges we have chosen two main types of base materials with low thermal conductivity (see Table 1): The first type was Poly-ether-ether-ketone (PEEK). It is a technical thermoplastic polymer qualified for space, ultra-high-vacuum, and cryogenic conditions. The second type were anodic oxides growth on Titanium[4] and Aluminum[5], being common structural materials for aerospace applications.

Table 1. Comparative thermal conductivities of some materials employed in this work and two common Al and Ti alloys.

THERMAL CONDUCTIVITIES	
Material	k ($W \cdot m^{-1} \cdot K^{-1}$)
PEEK[6]	0.22
TiO_2 nanotubes[7]	0.75-0.98
Porous Anodic Alumina[8]	1.5
Aluminum (6163-0)[9]	218.1
Titanium (Ti-6Al-4V) [9]	7.3

In this contribution, we will focus on the synthesis and preliminary characterization of some these nanostructured functional metamaterials that we propose to be included in future ground and space cryostats.

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2. SYNTESIS AND CHARACTERIZATION METHODOLOGY

We have synthesized and characterized the different nanostructured metamaterials following two parallel scalable synthesis strategies:

i) Conformal Self-ordered three-dimensional networks of nanowires (3DNN) obtained by electrochemical routes on Al.

ii) Nanocomposite flexible films based on PEEK matrix and a) Polyether-ether-ketone (PEEK) covered with Au nanocolumns (PEEK/Au); and b) filled with anodic TiO_2 nanotubes (PEEK/TiO_2) and FeNi thin films (PEEK/FeNi).

The techniques for surface characterization of the samples included Scanning Electronic Microscopy (SEM, Zeiss Supra 25) and Atomic Force Microscopy (AFM, Nanoscope). The functional characterizations consisted in the measurement of hysteresis loops and the frequency dependence[10] of the reflection and transmission coefficients parameters, from the magnetic susceptibility and electrical permeability. The hysteresis loops were measured by a Vibrating Sample Magnetometer (VSM, EZ-7 Microsense) and electromagnetic parameters were acquired by commercial set-up (Epsimu, Multiwave), see Fig. 1.

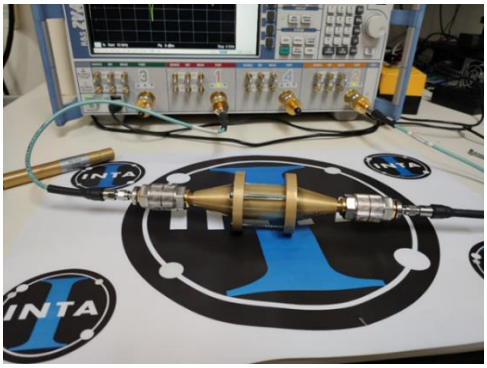


Figure 1 Set-up Epsimu for measuring magnetic susceptibility and electrical permeability

Synthesis of 3D Networks of Nanowires (3DNN)

The 3DNN synthesis relies on the anodization of Al for generating well-ordered porous alumina. The synthesis of 3DNN comprised a double DC anodization process, where the second process includes voltage pulses above hard anodization regime[5]. These pulses generate a modulation of the composition suitable for a selective chemical etching. As a result, each nanopore link to its six closer neighboring pores by transversal nanopores. These connections correspond to each pulse during the anodization, and thus multiple pulses generate multiple connections at precise periods (P). These networks of nanopores have been employed as templates for polymer infiltration and electroplating metallic materials[5, 8, 12]. Figure 2 presents SEM images of the porous alumina templates and 3DNN.

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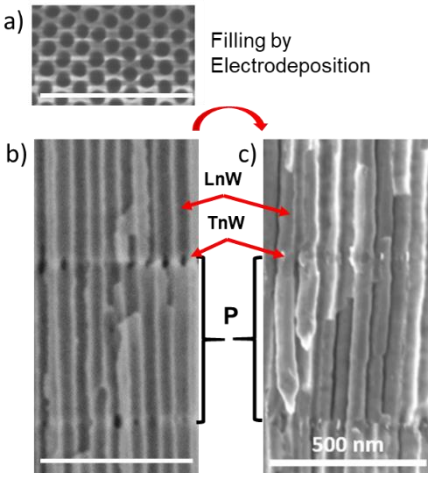


Figure 2 a) and b) Top and cross section SEM image of anodic porous alumina used as template. C) Cross section Co 3DNN image where Longitudinal Nanowires (LnW) (10's µm length, 55 nm diameter) separated 65 nm. The LnW interconnect by: Transversal nanowires (TnW) (diameter 35 nm) distributed in regular periods (P) of 100's nm. Scale bars are the same for all the images.

Synthesis of PEEK/Au nanocomposites

PEEK/Au composites (Au columns of 60 nm in diameter and 200 nm in length on PEEK) were synthesized by Nanostine©'s using its proprietary sputtering deposition process[11] on 0.01 mm thick PEEK films. Figure 2 shows a SEM image of the resulting surface of the flexible nanocomposite.

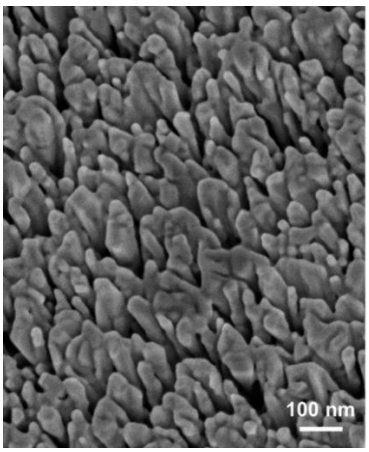


Figure 3 SEM image of PEEK/Au Surface

Synthesis of PEEK/TiO_2 nanotubes based nanocomposites.

The anodization of Ti foils in Ethylene Glicol/water/NH_4F electrolytes (50V) for 1 hour resulted in 11 µm thick homogeneous layers of TiO_2 nanotubes. After the anodization process, the TiO_2 nanotubes were annealed under N_2 atmosphere at 400 ºC for 1 hour. This annealing assured a metastable TiO_2 Anatase phase and the desorption of possible volatiles within the nanostructure. Once the annealing process finished, we performed the hot-embossing of PEEK[4, 11] at 365 ºC applying 25 kN and their removal from the Ti foil by a mechanical stripping. The resulting composites were employed for two different porpoises: i) The realization of magnetic nanocomposites by sputtering 15 nm thick $Ni_{80}Fe_{20}$ *Permalloy* (Py) on the nanostructured surface and, ii) The chemical etching of TiO_2 nanotubes generating nanostructured PEEK film. Fig. 4 shows a sketch of these synthesis processes with SEM and AFM images.

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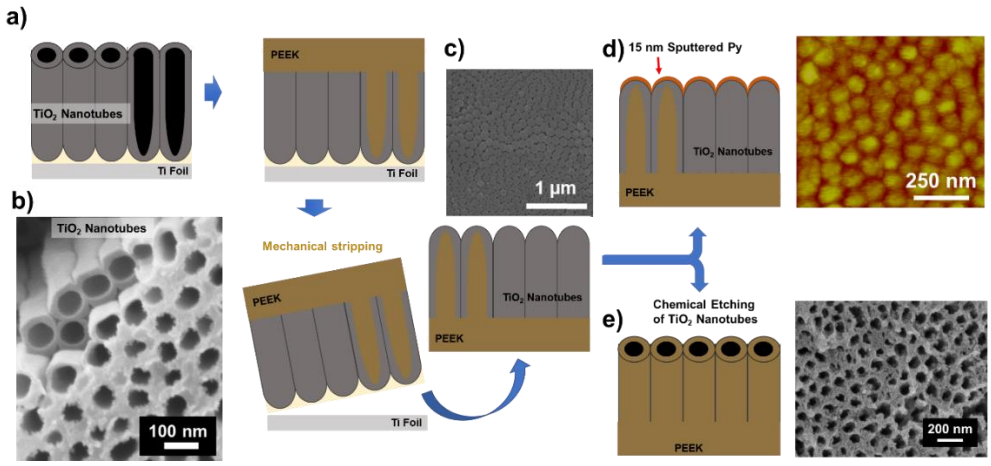


Figure 4 a) Sketched processes for the synthesis of TiO_2/PEEK based composites (adapted from [13]). b) SEM image of top surface from the as obtained TiO_2 Nanotubes c) Image of the resulting surface of TiO_2/PEEK composites (bottom of TiO_2 nanotubes) d) Sketch of Py on TiO_2/PEEK with AFM image and e) Sketch of nanostructured PEEK after chemical etching of TiO_2 nanotubes.

Functional Characterization

The magnetic characterization results of Cobalt 3DNN and Py on TiO_2/PEEK composites are presented in Figure 5 a) and b) respectively. Figure 5 a) shows the registered hysteresis loops applying magnetic field from in-plane (IP) to out-of-plane (OOP) orientation. The hysteresis loops present a clear magnetic anisotropy with an in-plane easy axis. The coercive field reaches above 0.02 T but still useful for DC magnetic shielding. However, the hysteresis loops of Py on TiO_2/PEEK in the same IP configuration exhibits 0.002 T of coercive field making it more adequate for low field DC shielding. Figure 5 c) shows the results of the electromagnetic characterization of PEEK/Au. The absorption coefficient presents evident peaks at 1.7 and 9.5 GHz with a behavior, both in reflectance and transmission coefficients, similar to that of a thin metal film.

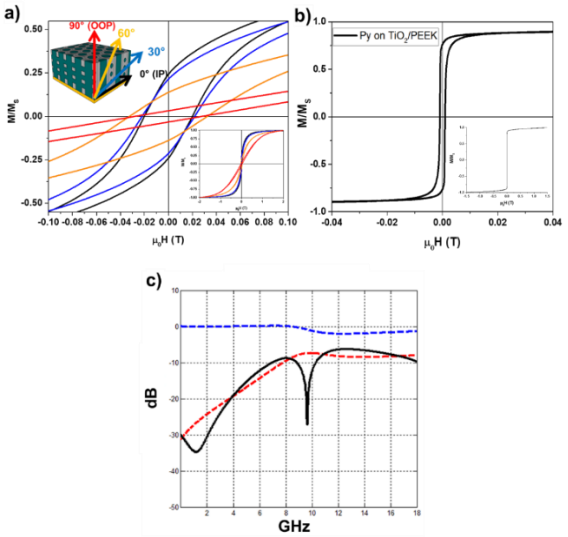


Figure 5 Room temperature magnetic hysteresis loops of: a) Co 3DNN b) Py on TiO_2/PEEK. c) Multiband characterization results of PEEK/Au nanocomposites. Coefficients: Transmission in blue, Reflection in red, Absorption black.

3. CONCLUSIONS

The employed synthesis routes for the fabrication of the studied nanostructured metamaterials are scalable. These metamaterials present useful properties for electromagnetic isolation. These metamaterials present no evident limitations for vacuum and cryogenic environments. Although the thermal conductivity of the employed matrices (PEEK, anodic porous alumina and TiO_2 nanotubes) is lower than those of Ti and Al, further qualification tests must be performed in order to determine their optimal application.

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