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Non-destructive interrogation techniques for highly heterogeneous composite materials

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14. ABSTRACT This proposal is concerned with the study of highly heterogeneous composite materials. We propose to develop mathematical models in order to explain certain features such as electric properties by using nondestructive techniques. In order to achieve the proposed objectives we have been working in the following problems: • Study of the effective permittivity and reflectivity: Four models for the effective permittivity of composite materials were considered: Efimov, Lorentz, homogenization and a probability measure model. We have analyzed and compared their behaviour on simulated data. Afterwards, the probability measure model was adopted to analyze the effective permittivity of multilayer materials using real data for the permittivity of each material layer. • Parameter estimation for characterization of multilayer materials: The estimation of modeling parameters were performed including layer interface locations, thermal diffusion and heat conduction coefficients. The problem of source determination was also addressed, where regularization methods were developed to improve the identification results. An error bound for the estimation was derived and the dependency of the solution on data was studied. • Transmission through materials: Radon-like transforms are used in computed tomography to identify different parts of the body when looking for abnormalities. Here we have studied them as a data transmission analysis tool since they could be useful to provide information on the properties of composite materials. The inversions of different transforms of this type were obtained. This is a main topic when studying them, since it is required for the identification of the different parts of the body that is being analyzed.			
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heterogeneous composite materials*

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1 Accomplishments

This proposal is concerned with the study of highly heterogeneous composite materials. We propose to develop mathematical models in order to explain certain features such as electric properties by using nondestructive techniques.

- Research Objectives:

Main research objectives proposed for this project were

- To obtain a description of the heterogeneity of the material.
- To develop an adaptive approach to determine the relevant parameters of a multilayer material (depth and electric parameters of each layer)
- To use a Radon transform in order to analyze transmission data which could provide information about the properties of the different layers contained in a composite material.
- To develop a technique in order to characterize the degree of surface roughness of a material.

In order to achieve the proposed objectives we have been working in the following problems:

- *Study of the effective permittivity and reflectivity:* Four models for the effective permittivity of composite materials were considered: Efimov, Lorentz, homogenization and a probability measure model. We have analyzed and compared their behaviour on simulated data. Afterwards, the probability measure model was adopted to analyze the effective permittivity of multilayer materials using real data for the permittivity of each material layer.
- *Parameter estimation for characterization of multilayer materials:* The estimation of modeling parameters were performed including layer interface locations, thermal diffusion and heat conduction coefficients. The problem of source determination was also addressed, where regularization methods were developed to improve the identification results. An error bound for the estimation was derived and the dependency of the solution on data was studied.
- *Transmission through materials:* Radon-like transforms are used in computed tomography to identify different parts of the body when looking for abnormalities. Here we have studied them as a data transmission analysis tool since they could be useful to provide information on the properties of composite materials. The inversions of different transforms of this type were obtained. This is a main topic when studying them, since it is required for the identification of the different parts of the body that is being analyzed.

- Technical approach

This proposal is concerned with the study of highly heterogeneous composite materials. Complex materials, including ceramic matrix composites (CMC's), are increasingly used in a wide range of applications including in components of high temperature engines [3], [12]. While these materials offer many advantages, the harsh environments they are subjected to can lead to severe degradation. Nondestructive evaluation techniques are needed for detecting and characterizing degradation in these materials so that they can be

safely used on high-performance aircraft for instance. In CMC's, air at high temperatures causes formation of oxides. When oxidation occurs the material becomes brittle which leads to catastrophic failure. Applying a mechanical load increases this effect. A cross section of a CMC composed of carbon or silicon carbide (SiC) fiber and SiC matrix is shown in Figure 1. One of our purposes is to study and analyze certain properties of

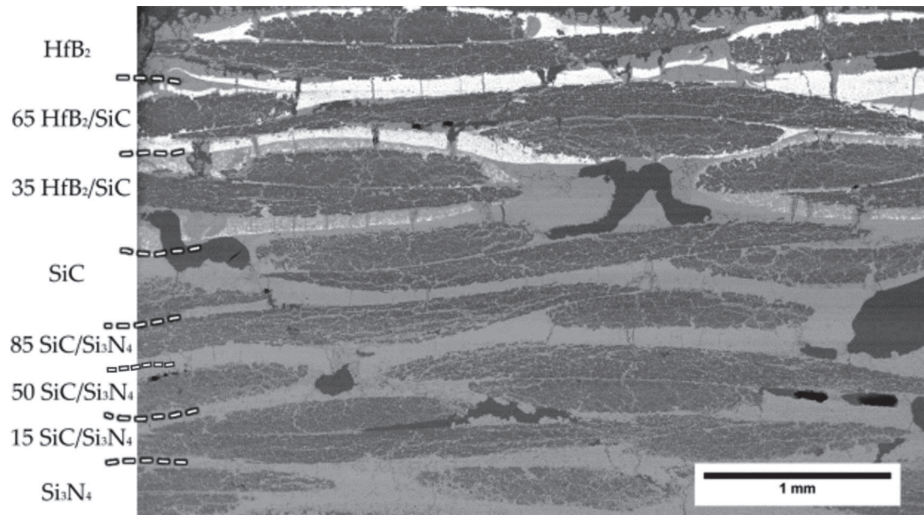


Figure 1: A section of a Ceramic Matrix Composite.

those materials such as electric and thermal properties. These results will be useful to characterize possible changes due to heat, stress, in their chemical composition using non-invasive and non-destructive interrogation techniques based on reflectance, absorption and transmission properties.

Matlab code is used for simulations. Some of them were developed by our group for each specific problem while the optimization processes were performed using functions provided by the software.

These efforts lead to the following accomplishments:

- 9 refereed publications in international journals, 1 of them in review process
- 12 refereed publications in proceedings and 1 technical report,
- 29 conference presentations.

To pursue our goals, we focused on three lines of work which provide complementary information:

Study of the effective permittivity and reflectivity.

We propose to estimate the effective permittivity as a function of the wavenumber using non-invasive and non-destructive interrogation techniques based on the reflectance coefficient.

The effective permittivity of a composite material is a macroscopic property that allows the material to be treated as if it were homogeneous and isotropic. A good effective permittivity model will allow us to identify changes in material properties caused by thermal sources. We use different mathematical models for the permittivity (Efimov, Lorentz, Probability distribution, homogenization) and compare their performances in the estimation process by numerically simulating noisy reflectance coefficients for a range of frequencies.

To simulate data we consider the recurrence formula (Orfanidis [13]) for the reflection responses at the successive interfaces of a multilayer material.

Each one of the four models that were considered has a number of parameters that need to be determined. Parameter estimation ([5], [16], [1]) is an important stage in mathematical modelling, since obtaining a good model depends largely on the values used for the parameters. Using information available in the literature as a reference, the adjustment is made through an optimization process based on data obtained in previous numerical simulations and/or experimental data ([6], [15], [18]). Here, we estimate the parameters by minimizing the square errors between the reflectance data and the simulated reflectance obtained from the model. Denoting by θ the parameters to be estimated, we look for the $\hat{\theta}$ as follows

$$\hat{\theta} = \arg \min_{\theta} |R(k_j) - R(k_j, \theta)|^2 \tag{1}$$

where $R(k_j)$ denotes the reflectance data and $y_j = R(k_j, \theta)$ the simulated reflectance (that depends on the parameter θ) obtained with a particular model.

We have been working on this line in collaboration with H.T. Banks (NCSU) and Amanda Criner (AFRL) until Dec. 2019.

Afterwards, real values of the refraction index for different glasses were taken from [10] to study the effective permittivity. We use the Prohorov metric framework model described in [4],

$$\hat{\epsilon}(k) = \epsilon_{\infty} - (\epsilon_s - \epsilon_{\infty}) \int \frac{k_0^2}{k^2 - ik/\tau - k_0^2} dG(k_0), \tag{2}$$

to estimate the effective permittivity $\hat{\epsilon}$ as a function of the wavenumber k , where ϵ_s and ϵ_{∞} denote the relative permittivity of the medium at zero frequency and “infinite”, respectively; τ denotes the relaxation time and G the probability measure.

The refraction index is given as a function of the wavenumber, for irregular distributed frequencies. So, an interpolation method [7] was applied to obtain the values on a regular partition of the range of frequencies. Comparisons of the resulting reflectivity and effective permittivity for different settings involving the same materials for the layers were performed.

Figure 2 (Left) shows the simulated reflectance (circles) of a material composed by Vitreous Germania (VG), Mixed Borate Glasses (MBG) 15.0 (Me₂ + MeO) · 85.0B₂O₃, and 36.0BaO·64.0B₂O₃ Glass (BBG). The layers are assumed of equal length, placed in the following order: MBG-BBG-VG. Then, these layers are repeated few times in the same order. In the same figure, we plot the corresponding reflectance of the *effective* material, that is an homogeneous material with the resulting effective permittivity (solid lines). Figure 2 (Right) shows the real part of the effective permittivity obtained for the six, nine and fifteen layered materials.

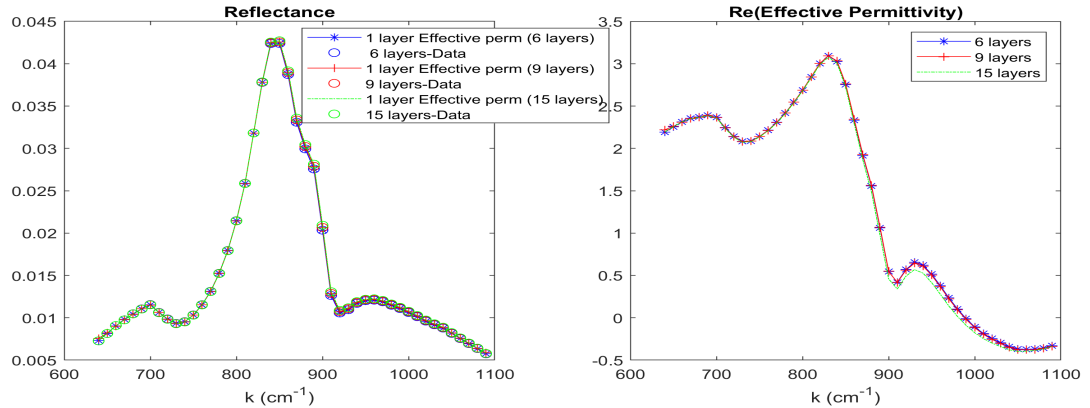


Figure 2: Left: Reflectance, Right: Real part of the Effective permittivity.

Accomplishments in this line of work:

- 4 oral presentations at national and international congresses.
- 1 technical report
- 1 referred publication in proceedings
- 1 paper (in review process)

Parameter estimation for characterization of multilayer materials.

As the exposure at high temperatures might lead to material degradation, we study the heat transfer problem in composite material and thermal source identification.

As a first approach we consider a 1D approach. Different settings were considered where a perfect assembly is assumed between the layers, hence the thermal resistance at the interface is neglected. Stationary heat process is considered to determine the parameters of the model. Differences between experimental and simulated temperature data would provide information about the imperfections at contact points. We want to point out that we are not modeling a real case where there are thermal resistance at layer interfaces. However, the discrepancy between experimental and simulated data should not be too much, otherwise important separations, cracks or imperfections at solid-solid interfaces are present at interfaces and must be analyzed.

The heat conduction process may be described by a parabolic partial differential equation with interface and boundary conditions. An analytical solution was obtained for a two-layer material. For the numerical simulation, Matlab was used, where the discretization

of the differential equations was faced using Crank-Nicolson finite difference method [7], centered on the spatial variable and forward in the temporary variable. This is an implicit method, frequently used in the numerical resolution of parabolic-type equations, such as the advective-diffusive-reactive transport equation, because it guarantees first-order precision in time and second-order precision in space [14]. It is also numerically stable under certain conditions [11],[17].

The thermal properties, such as thermal conductivity and diffusivity, are assumed to be piecewise constant functions. We have faced the problems of estimation of the different parameters in the model, including heat transfer, thermal conductivity and diffusivity coefficients for multilayer material. We have also studied the identification of thermal sources for a homogeneous material. By means of Fourier techniques it can be shown that the problem is ill-posed in the sense that the solution exists but it does not vary continuously with the data. We consider a family of parametric regularization operators to deal with the ill-posedness of the problem. We proposed a rule to select the regularization parameter as a function of noise level in data in order to obtain a regularized solution that approximates the unknown source. We find a Hölder type bound for the error of the approximated source when the unknown function is considered to be bounded in a given norm. Moreover, sensitivity analysis was performed to determine the dependency of the temperature on available data. Figure 3 shows the estimated sources and their curve levels obtained for non-regularized and regularized approaches.

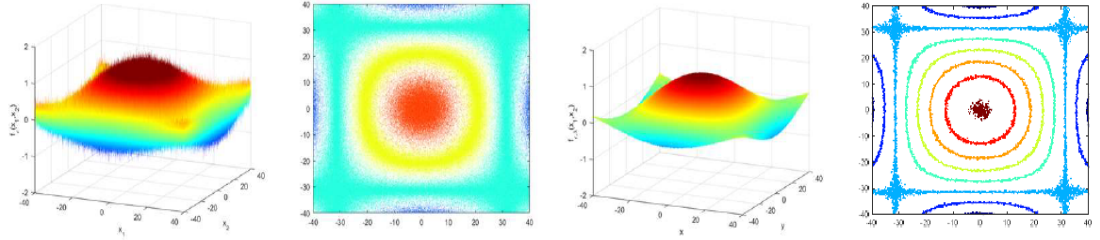


Figure 3: No-regularized (left) and regularized (right) estimated Source and its curve levels

Finally, effective thermal conductivity was obtained for multilayer materials based on the temperature value at the end point, say $u(L)$, where u is the temperature function, given by

$$\kappa_{ef} = \left[\sum_{i=1}^{M-1} \frac{l_i}{L} \left(\frac{1}{\kappa_i} - \frac{1}{\kappa_{i+1}} \right) + \frac{1}{\kappa_M} \right]^{-1}. \quad (3)$$

being κ_i , $i = 1, \dots, M$ the thermal conductivity coefficients of the material layer i for a multilayer material composed by M layers.

Accomplishments in this line of work:

- 13 presentations at national and international congresses.
- 6 referred publication in proceeding
- 7 refereed publications in international journals.
- 1 paper (in review process)

Transmission through materials

In this line of investigation we have made progress in different directions:

We studied inverse problems in the field of Transmission Compton Scattering Tomography (CST). This promising imaging technique consists in using an external source of radiation together with peripheral detectors in order to explore the inner parts of an object. Detectors are set to record scattered, i.e. non primary, photons, in a specific range of energy. This approach could provide a useful tool when looking for information about properties of the different layers contained in a composite material when data transmission is available.

The direct problem consists in modeling the data recorded by detectors whereas the inverse problem is how to recover the electronic density of the object under study.

Different setups, i.e. configurations of source and detector, have been proposed. Each one has its own advantages and drawbacks regarding the number of detectors, the need of using moving parts in the system, the required energy resolution, etc. Different setups lead to different geometries and so to different inverse problems. We have been studying two different systems where signal at detectors is modeled using Radon transforms either on circular arcs, in two dimensions, or on torus surfaces, in three dimensions.

In particular, the three dimensional system has a rather simple design: a fixed source S and just one detector D that moves on a spherical surface around the source. The most outstanding feature of this setup is its simplicity. The manifold of the corresponding Radon transform is a torus, so data is modeled by a Radon transform on tori.

We have proved interesting properties of the corresponding Toric Radon Transform including its invertibility. This result is important because it enables the search of an inversion method. In fact, an essential part of our work was the development and numerical implementation of the reconstruction algorithms, which allowed us to carry out simulations that support the theoretical results and show the potential of the methods studied in biomedical imaging applications and in other areas such as control non-destructive.

Accomplishments in this line of work:

- 8 presentations at national and international congresses.
- 4 referred publication in proceeding
- 1 refereed publications in international journals and 1 in review process.

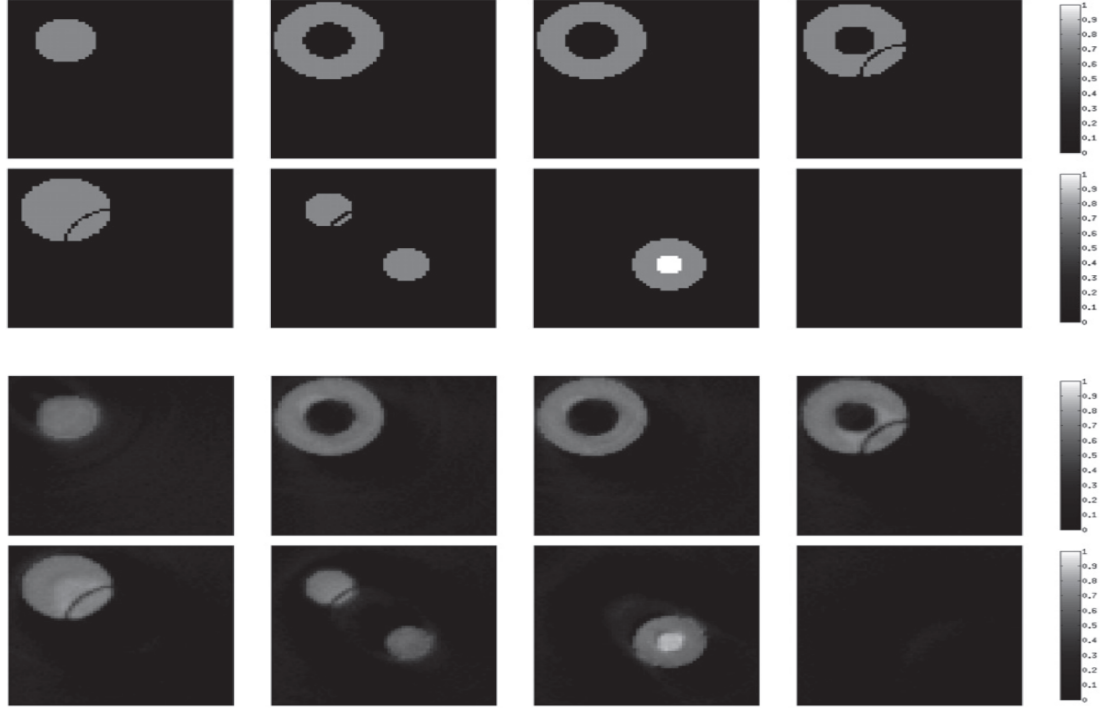


Figure 4: Top: Slices of Original 3D phantom. Bottom Reconstruction from noisy data using Tikhonov regularization

- Difficulties encountered:

- Due to the COVID pandemic, Argentine universities have been closed from March 2020 until the beginning of 2022. From one day to the other we couldn't get access to computers, programs or anything we needed for research. We had to start over an important part of the research work. On the other hand, it was assumed that Amanda Criner would provide data from the AFRL (Materials and Manufacturing Directorate, Air Force Research Laboratory WPAFB 45433, USA) but the lab has been closed for long time and we couldn't get experimental data as planned.
- In December 2019, our main collaborator, H. Thomas Banks, of the Center for Research in Scientific Computing (CRSC) at NSCU, passed away. His continued collaboration was extremely important because of his advice and input. In addition to the value of his suggestions and his great experience, it was also a great affective loss since we have lost an invaluable person and a friend.

These exceptional situations significantly altered our research schedule and we couldn't make progress on the last objective: To develop a technique in order to characterize the degree of surface roughness of a material.

In this objective, we have started to study the method of Empirical Mode Decomposition (EMD) to analyze the roughness of a surface. We are in the first stage of at this point, analyzing the method and studying algorithms that can be used towards the objective.

2 Impacts

2.1 Development of the principal discipline of the project

Algorithms and mathematical techniques were developed making a contribution for the study of modeling and parameter estimation, with particular application to multilayer materials. Also, existing regularization and mathematical inversion techniques were improved and/or extended to higher dimensional problems.

2.2 Other disciplines

The mathematical algorithms are not restricted to composite material, they can be applied to a wide range of problems arising in other disciplines like biology, chemistry or medicine that involve parameter estimation problems.

2.3 Describe the impact in this reporting period on the development of human resources

The project provided good opportunities to the graduate students to participate and assist to conferences and continuing their research activity since our country is having serious economic difficulties. Thus, the financial assistance for conference participation was very important.

3 Publications - Conference Presentations - Participation at Scientific Events

3.1 Publications journals

1. G. F. Umbricht; D. Rubio y C. El Hasi, A regularization operator for the source approximation of a transport equation. *Mecánica Computacional* 37(50) (2019), pp: 1993–2002. ISSN: 2591-3522.
2. G. F. Umbricht; D. Rubio, Rodolfo Echarri y Claudio El Hasi, A technique to estimate the transient coefficient of heat transfer by convection. *Latin American Applied Research* 50(3) (2020), pp: 229–234. ISSN: 0327-0793/1851-8796.
3. G. F. Umbricht; D. Rubio and D.A. Tarzia. Estimation technique for a contact point between two materials in a stationary heat transfer problem. *Mathematical Modelling of Engineering Problems* 7(4) (2020), pp: 607–613. ISSN: 2369-0739/2369-0747.
4. J. Cebeiro; C. Tarpau; M. Morvidone; D. Rubio; M K Nguyen On a three-dimensional Compton scattering tomography system with fixed source. *Inverse problems.*: IOP Publishing Ltd, vol. 37, n° 5, 2021.
5. Umbricht, Guillermo Federico; Rubio, Diana y Tarzia, Domingo Alberto. Estimation of a thermal conductivity in a stationary heat transfer problem with a solid-solid interface. *International Journal of Heat and Technology* 39(2) (2021), pp: 337–344. ISSN: 0392-8764.
6. Rubio, Diana; Tarzia, Domingo Alberto y Umbricht, Guillermo Federico. Heat transfer process with solid-solid interface: Analytical and numerical solutions. *WSEAS Transactions on Mathematics* 20 (2021), pp: 404–414. ISSN: 1109-2769/2224-2880.
7. Umbricht, Guillermo Federico y Rubio, Diana. Optimal estimation of thermal diffusivity in an energy transfer problem. *WSEAS Transactions on Fluid Mechanics* 16 (2021), pp: 222–231. ISSN: 1790-5087/2224-347X.
8. Umbricht, Guillermo Federico; Tarzia, Domingo Alberto y Rubio, Diana. Determination of two homogeneous materials in a bar with solid-solid interface. *Mathematical Modelling of Engineering Problems* 9(3) (2022), pp: 568–576. ISSN: 2369-0739/2369-0747.

3.2 Publications in technical reports and refereed proceedings

1. G.F. Umbricht; D. Rubio; D. Tarzia. Problemas inversos asociados a un proceso estacionario de transferencia de calor. *MACI* 7 (2019), pp 593-596.
2. M. Morvidone; D. Rubio. Notas sobre la estabilidad de la inversa de la transformada v en el plano. *MACI* 7 (2019), pp 421-424.
3. E. Porten, J.M. Medina, M. Morvidone, Algoritmo iterativo para la reconstrucción de una señal a partir de un muestreo aleatorio, *MACI* 7 (2019)
4. G. F. Umbricht; D. Rubio. Identificación de la fuente en una ecuación de transferencia de calor en un tejido biológico. *MACI* 7 (2019), pp 405-408.

5. H.T. Banks, Jared Catenacci, Amanda Criner, M. Morvidone, Diana Rubio, and N. Saintier. Comparison of methods for the determination of reflective properties of complex composite materials. Technical Report. CRSC-TR19-12, 2019.
6. J. Cebeiro, M. A. Morvidone, D. Rubio, C. Tarpau and M. K. Nguyen, "A new Transmission Compton Scattering Tomography," 2019 XVIII Workshop on Information Processing and Control (RPIC), 2019, pp. 259-264, doi: 10.1109/RPIC.2019.8882134.
7. E. Porten, J.M. Medina, M. Morvidone, Random Sampling over LCA groups and inversion of the Radon Transform, MACI VIII (2021)
8. Diana Rubio; Marcela Morvidone; Nicolas Saintier. Estimación de parámetros en materiales compuestos para el análisis de la corrosión provocada por altas temperaturas. MACI 8 (2021).
9. Guillermo F. Umbricht; Diana Rubio; Claudio El Hasi. Solución Analítica de un problema de transferencia de energía térmica, con generación de calor, disipación por convección y flujo lateral. MACI 8 (2021).
10. Guillermo F. Umbricht; Diana Rubio; Domingo A. Tarzia. Transferencia de calor estacionaria en materiales multicapa. MACI 8 (2021).
11. Guillermo F. Umbricht; Diana Rubio. Estimación del coeficiente del término difusivo en una ecuación parabólica completa. MACI 8 (2021).
12. Guillermo F. Umbricht; Diana Rubio; Domingo A. Tarzia. Determinación de los materiales que componen una barra con interfaz sólido-sólido. MACI 8 (2021).

3.3 Participation in scientific events

1. Erika Porten, Marcela Morvidone, Juan Medina. Algoritmo iterativo para la reconstrucción de una señal a partir de una muestra aleatoria. LXVII Reunión Anual de la Unión Matemática Argentina, UMA 2018, La Plata. September 2018.
2. Guillermo Umbricht and Diana Rubio. *Approximation of a source for a transport equation by means of a regularization method*. San Miguel de Tucumán. Argentina. XII Congreso Argentino de Mecánica Computacional (MECOM 2018). November, 2018.
3. Marcela Morvidone y Diana Rubio. Source Image Reconstruction from a Compton Camera Data, ICAMDC-MMMELS, Buenos Aires, Nov. 2018.
4. Guillermo F. Umbricht, Diana Rubio, Identificación de la fuente en una ecuación de transferencia de calor en un tejido biológico, VII Congreso de Matemática Aplicada, Computacional e Industrial. Río Cuarto, Córdoba, Argentina, May, 2019.
5. Guillermo Federico Umbricht, Diana Rubio, Domingo Tarzia Problemas inversos asociados a un proceso estacionario de transferencia de calor, VII Congreso de Matemática Aplicada, Computacional e Industrial. Río Cuarto, Córdoba, Argentina, May, 2019.
6. E. Porten, J.M. Medina, M. Morvidone, Algoritmo iterativo para la reconstrucción de una señal a partir de un muestreo aleatorio, VII Congreso de Matemática Aplicada, Computacional e Industrial. Río Cuarto, Córdoba, Argentina, May, 2019.

7. H.T. Banks; M. Morvidone; D. Rubio; N. Saintier. Comparison of methods for determining the effective permittivity of composite materials. International Congress on Industrial and Applied Mathematics. Sociedad Española de Matemática, International Council for Industrial and Applied Mathematics, Valencia, España, Julio 2019.
8. J. Cebeiro, M. A. Morvidone, D. Rubio, C. Tarpau and M. K. Nguyen, *A new Transmission Compton Scatter Tomography based on Backscattered Radiation*, XVIII Workshop on Information Processing and Control (RPIC), Bahía Blanca, Argentina, Sept. 2019.
9. Guillermo F. Umbricht, Diana Rubio Método de suavización para la identificación de la fuente en una ecuación de transporte. LXVIII Reunión anual de Comunicaciones Científicas en el I Encuentro Conjunto de la Sociedad Matemática de Chile y la Unión Matemática Argentina. Universidad Nacional de Cuyo, Mendoza, Mendoza, Argentina. Sept. 2019.
10. J. Cebeiro, M. A. Morvidone, D. Rubio, C. Tarpau and M. K. Nguyen, Transformada de Radon sobre un par de arcos de círculo. LXVIII Reunión anual de Comunicaciones Científicas. I Encuentro Conjunto de la Sociedad Matemática de Chile y la Unión Matemática Argentina. Mendoza, Mendoza, Argentina. Sept. 2019.
11. Guillermo F. Umbricht, Diana Rubio *Método de suavización para la identificación de la fuente en una ecuación de transporte*. LXVIII Reunión Anual de Comunicaciones Científicas. Mendoza, Argentina. Sept. 2019
12. Guillermo F. Umbricht, Diana Rubio, Rodolfo Echarri, Claudio D. El Hasi. Sobre una estimación del coeficiente de transferencia de calor por convección en régimen transitorio. estudio numérico de elasticidad. 104^a Reunión de la Asociación Física Argentina, Santa Fe, September 30 - October, 3, 2019.
13. G. Umbricht, D. Rubio and C. El Hasi. A regularization operator for the source approximation of a transport equation. XXIV Congreso sobre métodos numéricos y sus aplicaciones. Santa Fe, Argentina. Nov. 2019.
14. Diana Rubio. Estimación de parámetros para la modelización matemática. Aplicación al análisis de propiedades en materiales compuestos, (virtUMA) . LXVIV Reunión Anual de Comunicaciones Científicas de la UMA. Virtual Sept. 2020.
15. Erika Porten, Juan M. Medina, Marcela Morvidone, Random Sampling over LCA groups and inversion of the Radon Transform, VII Congreso de Matemática Aplicada, Computacional e Industrial. Virtual. La Plata, Argentina, May, 2021.
16. Diana Rubio; Marcela Morvidone; Nicolas Saintier. Estimación de parámetros en materiales compuestos para el análisis de la corrosión provocada por altas temperaturas. VII Congreso de Matemática Aplicada, Computacional e Industrial. Virtual. La Plata, Argentina, May, 2021.
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19. Guillermo F. Umbricht; Diana Rubio. Estimación del coeficiente del término difusivo en una ecuación parabólica completa. VII Congreso de Matemática Aplicada, Computacional e Industrial. Virtual. La Plata, Argentina, May, 2021.
20. Guillermo F. Umbricht; Diana Rubio; Domingo A. Tarzia. Determinación de los materiales que componen una barra con interfaz sólido-sólido. VII Congreso de Matemática Aplicada, Computacional e Industrial. Virtual. La Plata, Argentina, May, 2021.
21. Erika Porten, Juan Medina, Marcela Morvidone Inversion from a random sampling of the Radon transform in the context of LCA groups Mathematical Congress of Americas (MCA21), Buenos Aires, July 2021.
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