

Workshop on Shape Memory Alloys Processing, Properties and Applications CENIMAT, FCT/UNL, 19/02/2020



Severe plastic deformation

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Laser welding



x B2 + B19' # R

In situ study of thin film growth (sputter)





Localized treatment Gunctional gradient

Strain monitoring



Endodontic files / Fatigue fracture



Orthodontic archwire



In situ study of thermomechanical process







PROGRAMA

Apresentações orais

Apresentações orais						
Autor(es)	Instituição	Assunto	Hora			
F.M. Braz Fernandes	CENIMAT	Abertura. Apresentação do projecto FIBR3D	10h00 - 10h10			
F.M. Braz Fernandes	CENIMAT	SMA@CENIMAT	10h10 – 10h30			
JP Oliveira, B Crispim, Z Zeng, T Omori, FMB Fernandes, RM Miranda	UNIDEMI Univ.Electronic Science & Tech- nology-China Tohoku Univ. CENIMAT	Microstructure and mechanical Properties of gas tungsten arc welded Cu-Al-Mn shape memory al- loy rods	10h30 - 10h50			
Rafaella Magalhães	SPA da Boca	Fios de Níquel-Titânio na perspectiva da Medicina Dentária/Ortodontia	10h50 - 11h10			
Coffee Break / Posters						
Patrícia F. Rodrigues, F.M. Braz Fernandes, Andersan dos Santos Paula, Edgar Camacho, Patrick Inácio, Rafaella Magalhães, Telmo Santos, N. Schell	CENIMAT CEMPRE IME SPA da Boca UNIDEMI DESY	Functionally Graded Orthodontic Archwires	11h40 - 12h00			
P. Inácio, Telmo G. Santos, J.P Oliveira, E. Camacho, F.M. Braz Fernandes, N. Schell	UNIDEMI CENIMAT DESY	Produção e caracterização multi-fenómeno de fitas de ligas com memória de forma com gradiente funcional	12h00 - 12h20			
	12h20 - 14h20					
Jorge Martins António Ginjeira Duarte Marques Rui F. Martins F.M. Braz Fernandes	FMD-UL UNIDEMI CENIMAT	Premium brands versus Replica-like brand endo- dontic files	14h20 - 14h40			
Filipe Santos	DEC	Superelastic Tensegrities	14h40 - 15h00			
	15h00 - 15h30					
F.M. Braz Fernandes	CENIMAT	ak / Posters Conclusões. Encerramento.	15h30 - 15h45			

	Posters		
E. Camacho, Telmo G. Santos, P. Inácio, F.M. Braz Fernandes		Production and characterization of func- tionally graded wires from NiTi shape memory alloys	P1
E. Camacho, F.M. Braz Fernandes, P.F. Rodrigues, N. Schell		Thermomechanical Behaviour Of Shape Memory Rivet - In Situ Study	P2
Inês Marcelino, A. Velhinho, F.M. Braz Fernandes	CENIMAT	Additive Fabrication of Anepectic Meshes controlled by a NiTi alloy	P3
P.F. Rodrigues, P. Cunha, A.S. Ramos, J. Domingos da Costa, F.M. Braz Fernandes, M.T. Vieira	CEMMPRE IFBA CENIMAT	Using NiTi wires as crack sensors	P4
P.F. Rodrigues, Rafaella Magalhães F. M. Braz Fernandes	CENIMAT	In situ studies of NiTi and NiTiCu ortho- dontic wires	P5
J.O. Cardoso, E. Camacho, A. Velhinho, F. M. Braz Fernandes	CENIMAT	Surface roughness assessment with an optical microscope	P6
Filipe A. Santos, J. O. Cardoso, Edgar Camacho, A. Velhinho, F.M. Braz Fernandes, A. Micheletti		Modeling functional gradient in shape memory alloy wires	P7
A.R. Alves, F.M. Braz Fernandes	CENIMAT	Heat treatment of endodontic files	P8
F.M. Braz Fernandes, E. Camacho, C. Marcu, C. Gurau, G. Gurau	CENIMAT Univ Galati	Shape memory alloy-based composites fabricated by severe plastic deformation	P9
Andersan S Paula, Rebeca V Oliveira, Eduardo H Sallet, Danilo A C Gonçalves, Naiara V Lé Sénechal, Edilainea A O Melo, Rodolfo S Silva, Patrícia F Rodrigues, Luiz Paulo M Brandão, Jorge V L Silva, Paulo Inforçatti, Daniel L Bayerlein	Campinas	Produção de peças do sistema Ni-Ti-X via fusão seletiva a laser com mistura de	P10
M. Nascimento, P. Inácio, T. Paixão, S. Novais, E. Camacho, F. M. B. Fernandes, T. G. Santos, J. L. Pinto	DF Univ Aveiro, UNIDEMI CENIMAT	Monitorization of polymeric parts fabri- cated by 3D Additive Manufacturing and reinforced with NiTi wires by integrated optical fiber sensors	P11

ORAL PRESENTATIONS

SMA at CENIMAT

F.M. Braz Fernandes, Rui M.S. Martins, K.K. Mahesh, R.J.C. Silva, A.S. Paula, F. Neves, J. Brito Correia, S.V. Correia, R. Magalhães, P. Fernandes, E. Camacho, P. Inácio, T. Santos

A general overview of 15 years of Activity on shape memory alloys is presented, covering:

- In situ studies of the growth of NiTi thin films,
- Thermomechanical processing,
- Severe plastic deformation,
- Applications in dentistery (orthodontics and endodontics),
- Powder metallurgy,
- Functionally graded SMA

Microstructure and mechanical Properties of gas tungsten arc welded Cu-Al-Mn shape memory alloy rods

JP Oliveira 1, B Crispim 1, Z Zeng 2, T Omori 3, FMB Fernandes 4, RM Miranda 1,

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2 School of Mechanical and Electrical Engineering, University of Electronic Science and Technology of China, China

3 Department of Materials Science, Graduate School of Engineering, Tohoku University, Japan

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Large diameter rods of Cu-Al-Mn shape memory alloy were gas tungsten arc welded. The microstructural evolution was studied by electron microscopy techniques and its impact on the mechanical and functional response of the welded joints was assessed. The fusion zone exhibited a mixture of α and β phases, while the base material was composed only by the parent β phase. The refined grain structure of the fusion zone increased the material ductility and can improve the functional fatigue resistance of the welded joint when compared to the original base material.

Fios de Níquel-Titânio na perspectiva da Medicina Dentária/Ortodontia

Rafaella Magalhães

Functionally Graded Orthodontic Archwires

Patrícia Freitas Rodrigues, Francisco M. Braz Fernandes, Andersan dos Santos Paula, Edgar Camacho, Patrick Inácio, Rafaella Magalhães, Telmo Santos, Norbert Schell.

Conventional NiTi orthodontic arch wires generate constant forces in a wide range of displacement during orthodontic treatment. In order to have different forces of actuation in the incisive region (lower) and in the molar region (higher), localized heat treatment using Joule heat effect have been proposed. This study presents the characteristic of the functional gradient introduced in a commercial superelastic Ni-Ti orthodontic arch wire by localized heat treatment, at 300 °C during 10 min. The DSC measurement, synchrotron radiation-based X-ray diffraction (SR-XRD), and tensile test indicated the existence of functional gradient and the stress induced martensitic transformation evolution beginning at the center of the heat-treated segment.

Produção e caracterização multi-fenómeno de fitas de ligas com memória de forma com gradiente funcional

P. Inácio, Telmo G. Santos, J.P Oliveira, E. Camacho, F.M. Braz Fernandes, N. Schell

Fitas de NiTi laminadas a frio foram processadas através de tratamentos térmicos localizados por efeito de Joule produzindo um gradiente de funcionalidade no seu comprimento. Foi realizada uma sequência de três tratamentos em cada fita a 300, 350 e 400 °C com duração de 10 minutos cada, espaçados por segmentos não tratados. Procedeu-se à caracterização do gradiente de funcionalidade produzido através de uma caracterização multi-fenómeno, nomeadamente, caracterização térmica por calorimetria diferencial de varrimento medição de temperatura com câmara termográfica; medição da condutividade elétrica com sonda de 4-pontos e correntes induzidas; caracterização mecânica com a medição do campo de extensão durante ensaio de tração uniaxial; e caracterização estrutural com difração de raio-x. Foi possível verificar o efeitos dos tratamentos térmicos com as diversas técnicas, obtendo-se um perfil do gradiente de funcionalidade no comprimento das fitas. Assim, os avanços apresentados neste trabalho permitem a produção de tratamentos térmicos localizados sem a necessidade de recorrer a equipamentos mais dispendiosos.

Premium brands versus Replica-like brand endodontic files.

Jorge Martins, António Ginjeira, Duarte Marques, Rui F. Martins, F.M. Braz Fernandes FMD-UL, UNIDEMI, CENIMAT

With the advances in the metal alloy technology, it was possible to create a new concept of endodontic instruments. The introduction in Endodontics of the nickel-titanium mechanized files has changed the former instrumentation protocols, and created new ones, which revolutionized the way the root canal system instrumentation was being performed. One of the major concerns related with the root canal system mechanical preparation is the possibility of having an unexpected instrument fracture. Two types of nickeltitanium file modes of failure have already been identified and defined as torsional failure or cyclic fatigue failure.

The main goal of the present research project is to compare mechanized endodontic files from premium brands with similar (replica-like) instruments from other brands. The main question to be answered may be presented as: Are the replica-like instruments similar to the premium brand ones?

Superelastic Tensegrities

Filipe Santos

DEC

Superelastic tensegrity systems are prestressed structures composed by bars and cables in which some cables are realized with superelastic shape-memory alloys. These systems combine the peculiar features of tensegrity structures with those of shape-memory alloys and are particularly suitable for adaptive and variable-geometry systems. The main goal of this work is the design of systems with antagonistic actuation, that is to say, systems where two sets of superelastic cables can be actuated against each other in a reversible way.

POSTERS

Production and characterization of functionally graded wires from NiTi shape memory alloys

E. Camacho, P. Rodrigues, F. M. Braz Fernandes, P. Inácio, T. G. Santos, N. Schell

The present work aims for the production of controlled functionally graded NiTi through localized heat treatments, taking advantage of its high sensitivity to precipitation. Through heat treatment by Joule effect(JE), it is possible to create a localized heat treatment (HT) on small segments of NiTi Ni-rich wires. DSC, thermomechanical and in-situ XRD during mechanical tests were performed. With this localized heat treatment it was possible to establish a gradient of matrix compositional changes along the wire, producing differentiated thermo-mechanical behavior of the wire. Also, the reproducibility of the same heat treatment performed on various wires has been checked with DSC tests.

Shape memory rivets

E. Camacho, F.M. Braz Fernandes, P.F. Rodrigues, N. Schell

A Ti-rich NiTi shape memory alloy (SMA) was used to join two components through shape memory effect (Fig. 1) adapted from the principle presented by a recent patent [1] that opens interesting perspectives in the field of aeronautics. In the concept study and viability of such type of rivet, DSC, dilatometric and in-situ XRD during thermomechanical cycles were performed [2]. In situ XRD study during thermomechanical cycle was conducted in a modified dilatometer DIL-805, Bähr (Fig. 2)) at the HZG beamline (HEMS/P07-EH3, Petra III, DESY, Hamburg) to identify the structural changes following combined thermal and mechanical loading, namely the preferential variants orientation at different steps of the process.

Additive Fabrication of Anepectic Meshes controlled by a NiTi alloy

Inês Marcelino, F.M. Braz Fernandes, Alexandre Velhinho

An anepectic material is a metamaterial which simultaneously exhibits a negative coefficient of thermal expansion (CTE) and a negative Poisson coefficient. These meshes may be applied in situations where a response to a thermal stimulus is desired, such as in the aerospace and medical fields. In the current work, anepectic composite meshes, made from ABS and NiTi alloys, were fabricated with the aid of an additive manufacturing technique and subsequently characterized. Seven different mesh designs or material combinations were tested. In every case, a common passive part consists of ABS. A complementary active part, consisting of NiTi wire, differed from case to case, whether in terms of the particular behavior demonstrated (shape memory effect [SME] an/or superelasticity [SE]), the temperature of the relevant phase transformation, the geometry adopted, or the diameter used. All meshes were tested in a silicone bath, and their CTE was measured. The results showed that, under careful parameter selection it is possible to achieve an anepectic effect by combining ABS with SME or SE wires. The mesh that showed to have a more negative CTE (-3008 x 10-6 °C-1) combined SME wires with SE wires. With such combination, it was possible to activate the mesh bellow the glass transition of the polymer, at 38°C. For one of the seven fabricated meshes, cyclic tests of three heating and cooling were performed. But only during the first cycle could the anepectic behavior be preserved, the CTE becoming positive on the remaining cycles. Finite element simulation was also performed, where both positive and negative mesh displacements were verified.

Using NiTi wires as crack sensors

Patrícia Freitas Rodrigues¹, Pedro Cunha², Ana Sofia Ramos¹, José Domingos da Costa¹, Francisco Manuel Braz Fernandes³, Maria Teresa Vieira¹

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³CENIMAT – Departamento de Ciências dos Materiais, Universidade Nova de Lisboa – FCT/UNL – Portugal

The development of NiTi crack sensors is being carried out in the aim of the project CrackFree – Towards self-repairing metallic alloys. The project aims to explore a novel approach to detect (micro)cracks in metallic materials – sensor; and, subsequently, induce crack closure – actuator. This approach may contribute to repair damage in aerospace/aeronautical components, as well as in biomedical devices. In this context, NiTi SMAs wires can be used to detect cracks in metal matrixes. The stress-induced martensitic transformation of NiTi SMAs and the different resistivity of austenite and martensite are the basis for the "NiTi crack sensors".

In situ studies of NiTi and NiTiCu orthodontic wires

Patrícia Freitas Rodrigues, Francisco Manuel Braz Fernandes, Edgar Camacho, Rafaela Magalhães, N. Schell

In this study, a class of NiTiCu orthodontic wires thermal activitied was analyzed. This wire (CuNiTi Thermocopper NiTi – 35°C/Morelli) has 6%Cu. Microstructural characterization of the wire was performed using Differential Scanning Calorimetry (DSC), in situ synchrotron-based x-ray diffraction (SXRD) and three-point flexion test was performed by TMA analysis in the temperature range (05 to 40 °C). This study provided a better understanding of the behavior of these wires.

Surface roughness assessment with an optical microscope

J. O.Cardoso, E. Camach0, A. Velhinho, F. M. Braz Fernandes

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Improved adhesion between NiTi wires and polymer matrices constitutes a fundamental condition for successful additive manufacturing of composite parts using these materials as constituents. A possible strategy consists of creating crevasses at the surface of the metallic wires to promote anchoring to the matrix. To do so, it is possible to chemically attack the surface with an acid solution; however, the combined parameter variations allowable (e.g. acid concentrations and duration of exposure) are liable to result in a vast array of surface profiles warranting optimization.

Under the aegis of the FIBR3D research project (POCI-01-0145-FEDER-016414), the present work deals with the development of a simplified method to analyse the surface roughness of the treated metallic wire surfaces in a reliable and timely manner through optical microscopy. Given that the use of a profilometer would take too much time, the authors resorted to measure the relevant roughness parameters through a Leica DMI 5000 M optical microscope (OM) capable of reconstructing a vertically correlated image through integration of a series of images of the same Region of Interest obtained with different focal planes. The relevant parameters evaluated with this technique were the arithmetic mean (RA), root mean square (RMS), maximum valley depth (Rv), maximum peak height (Rp), maximum height of the profile (Rt), together with the distances between consecutive peaks and consecutive valleys.

To verify the reliability and accuracy of the optical measurement technique, its results were compared to those obtained with a profilometer over calibrated Vickers indentations performed over the surface of a high purity soft copper surface.

Thus, simply requiring a set of optical imaging acquisitions, a series of statistically robust measurements may be obtained in order to quantify the surface roughness of the chemically treated NiTi wire surfaces.

Modeling functional gradient in shape memory alloy wires

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Shape memory alloys (SMA) present interesting functional characteristics (shape memory effect and superelasticity) that make them quite attractive for a wide range of applications. These functional characteristics are a consequence of phase transformations that take place within well-defined temperature or stress ranges, depending on being thermal- or stress-induced. These temperature/stress ranges are functions of chemical composition and heat treatment of the material. For applications requiring a wider controllable range, a wider temperature/stress range than that associated to a specific composition/heat treatment may be required. In such a situation, the possible solution will be to use a functionally graded material. The purpose of this work is to provide a basic design methodology for functionally graded shape memory.

The purpose of this work is to provide a basic design methodology for functionally graded shape memory alloy wires. The evolution of the wires subjected to load and temperature changes is simulated by integrating a simple system of ordinary differential equations through standard numerical routines.

To describe the constitutive behavior of SMA cables, we choose the Tanaka–Voigt model since it can easily be implemented and adjusted to a wide set of experimental data. A benchmark of our procedure is presented on a three-element system simulating a functionally graded SMA wires.

We consider only SMA elements with superelastic behavior, i.e. they are in austenitic phase at ambient temperature. Furthermore, we assume that martensitic phase transformations induce

negligible temperature changes in SMA elements, because of the limited strain rates associated with small nodal velocities.

Heat treatment of endodontic files

F. M. Braz Fernandes, Ana Rita Alves, A. Machado, J. P. Oliveira

Three brands of rotary nickel-titanium endodontic instruments with some comparable geometric features but with different cross-section (similar tip size and same taper of .04) were selected for this study: MTwo .04(35) (VDW GmbH, Germany), K3 .04(30) (SybronEndo, Mexico) and K3XF .04(30) (SybronEndo, Mexico). K3XF

is made from novel R-phase heat-treated metal alloy while all other files were made from traditional Ni-Ti alloy. The instruments were analyzed under the following conditions: i) as-received (AR), ii) heat treated at 350°C, iii) heat treated at 400°C. The transformation temperatures were determined by differential scanning calorimeter (DSC). Compared to conventionally K3 and Mtwo files, R-phase heat treatment K3XF file showed higher transformation temperatures. These results also showed that heat treatments increase the transformation temperatures.

Shape memory alloy-based composites fabricated by severe plastic deformation.

F.M. Braz Fernandes, E. Camacho, C. Marcu, N. Schell, C. Gurau, G. Gurau High-Speed High Pressure Torsion (HS-HPT) has been used to promote severe plastic deformation of Nirich and Ti-rich Ni-Ti shape memory alloys, fabricating multiple layered structures (up to 32 layers), embedding other metallic alloys, such as Mg alloy. The phases present at room temperature were identified by synchrotron radiation based X-ray diffraction. The transformation characteristics were determined by Differential Scanning Calorimetry (DSC) using a series of thermal cycles comprising 2 heating ramps up to 500°C intercalated by 3 conventional cooling / heating cycles between +150 and -150 °C in order to assess the relevance of recrystallization and precipitation phenomena.

Produção de peças do sistema Ni-Ti-X via fusão seletiva a laser com mistura de pós individuais de alta pureza

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A manufatura aditiva (Impressão 3D) consiste em um método de fabricação que obtém peças através da construção sucessiva de camadas, umas sobre as outras, obtendo, por fim, um objeto composto de camadas sobrepostas. Essa tecnologia está se desenvolvendo desde a década de 1980, entretanto obteve um desenvolvimento mais acelerado após os anos 2000 [1, 2], com o crescimento da capacidade dos softwares, da mecatrônica e da ciência dos materiais. Neste projeto de pesquisa será analisada uma técnica de manufaturas aditiva denominada Fusão Seletiva a Laser (FSL) para a produção de peças de liga do sistema Ni-Ti-X, com foco inicial em ligas binárias de NiTi, a partir de mistura de pós individuais de alta pureza. Uma limitação deste método de produção decorre da anisotropia advinda do próprio método de fabricação. As pecas fabricadas através de manufatura aditiva apresentam comportamentos mecânicos bem distintos quando submetidas a esforços nas direções paralelas e perpendiculares as camadas depositadas na construção da peca [3]. A grande aplicação tecnológica deste trabalho é tornar possível a obtenção de pecas com as propriedades especificas das ligas de níquel-titânio através do uso da manufatura aditiva. Este método de produção de peças ainda não ocupa grande percentual na escala global de produção, mas com um grande potencial de crescimento e capacidade de revolucionar a cadeia de produção de bens manufaturados [4]. A logística de produção possivelmente será reinventada a fim de garantir equipamentos e suprimentos de máquinas de manufatura aditiva. O objetivo principal do projeto, portanto, é encontrar a potência e densidade de energia ideais do laser a ser utilizado na FSL, com uso de pós de alta pureza produzidos em empresas ou institutos de pesquisa brasileiros. Trabalha-se com a premissa de utilização de pós individuais de cada metal, logo o sucesso da obtenção da liga metálica do sistema Ni-Ti-X (NiTi equiatômico e uso de Cu ou Nb para as ligas ternárias) reside na mistura eficiente dos pós sólidos de alta pureza e na boa interdifusão dos dois (ou três) metais durante a fusão dos mesmos pelo laser.

Monitorization of polymeric parts fabricated by 3D Additive Manufacturing and reinforced with NiTi wires by integrated optical fiber sensors

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Optical fiber sensors has been recently proposed on detection of defects in Additive Manufacturing (AM) systems. There are many advantages associated with the fiber sensors technology, such as reduced dimensions, chemical inertness, multiplexing capability, nearly punctual operation, and possibility to measure different parameters within one single optical fiber. Regarding the specific application for AM processes, these sensors can simultaneously measure temperature and strain induced.

This work focuses on three main issues regarding Material Extrusion (MEX) AM of thermoplastic composites reinforced by pre-functionalized continuous Nickel-Titanium (NiTi) wires: (i) evaluation of the effect of the MEX process on the properties of the pre-functionalized NiTi; (ii) evaluation of the mechanical and thermal behavior of the composite material during usage; (iii) the inspection of the parts by Non-Destructive Testing. For this purpose, an optical fiber sensing network, based on fiber Bragg gratings and a cascaded optical fiber sensor, was successfully integrated during the 3D printing of a polylactic acid (PLA) matrix reinforced by NiTi wires. Thermal and mechanical perturbations have been successfully registered as a consequence of thermal and mechanical stimuli.

The experimental results shown the good adhesion of the cascaded optical sensor to the surrounding material, where a maximum contraction of ≈100 µm is detected by the cascaded sensor in the PLA material at the end of the heating step (induced by Joule effect) of NiTi wires and a thermal perturbation associated to the structural transformation of austenite to R-phase is observed during the natural cooling step, near 33.0 °C. Regarding tensile cycling tests, during the unload step, a slope change in the temperature behavior is detected, which is associated with the material transformation of the NiTi wire (martensite to austenite). The integrated optical sensing methodology proved to be an efficient and precise tool to identify structural transformations regarding the specific application as a Non-Destructive Testing for AM.