## PROCEEDINGS

### Spring School for Young Researchers New Trends in Experimental Mechanics - NTEM 1

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## **INVITED LECTURES**





#### Experimental mechanics at temperatures close to absolute zero

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Experimental mechanics at extremely low temperatures is of particular interest for the superconducting and space technologies. Metals and alloys characterized by the fcc lattice are massively used in cryogenic applications. Many of these materials demonstrate excellent physical and mechanical properties in the proximity of absolute zero [1]. Depending on the material, various phenomena may occur at cryogenic temperatures, i.e. intermittent (discontinuous) plastic flow, plastic strain induced fcc-bcc phase transformation, evolution of microdamage, as well as low temperature creep and fracture. In the light of the thermodynamic instability, characteristic of weakly excited lattice and resulting from 3<sup>rd</sup> principle of thermodynamics, the thermo-mechanical coupling strongly affects deformation of materials operating at the temperatures close to absolute zero.

Experimental identification of the plastic strain induced phenomena in the near 0 K regime is complex. It involves unique set-up (Fig. 1) consisting of a double-wall vacuum insulated cryostat, well instrumented insert containing the specimen, a transfer line linking liquid helium (or liquid nitrogen) dewar with the cryostat mounted in the loading device. Moreover, an extended set-up for complex loads, to perform the non-proportional loading paths, is used. The experimental methods stretch from testing the materials inside the cryostat, via the magnetometric techniques, the acoustic emission, the scanning electron microscopy with the electron backscatter diffraction analysis, until the use of a source of synchrotron radiation. All principles of experimental mechanics are successfully applied.



Figure 1: The cryogenic set-up, the cryostat and the scanning electron microscope (SEM, EBSD)

Acknowledgments: grant of National Science Centre 2021/41/B/ST8/01284 is acknowledged

[1] R. Ortwein, B. Skoczeń, J.Ph. Tock (2014). Micromechanics based constitutive modeling of martensitic transformation in metastable materials subjected to torsion at cryogenic temperatures, International Journal of Plasticity, 59, 152-179.





## Characterization and modeling of materials applied in cryogenic conditions

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Design of modern scientific instruments (particle accelerators, detectors, superconducting magnets, NMRs, etc.), operating in the superconducting regime, involves constitutive modeling of materials applied at extremely low temperatures. It comprises three coupled dissipative phenomena: intermittent plastic flow (IPF), plastic strain induced fccbcc phase transformation and evolution of micro-damage, including radiation induced damage. The IPF (serrated yielding) represents oscillatory mode of plastic deformation [1]. Multiscale constitutive model of IPF involves microscopic approach based on the analysis of evolution of dislocations density and formation of dislocation pile-ups on the lattice barriers. Moreover, massive formation of twin boundaries is also taken into account. The plastic strain induced phase transformation consists in rapid change of crystallographic structure from face centered cubic (fcc) to body centered cubic (bcc). The constitutive model takes into account the micro-mechanical phenomena, such as the interaction of dislocations with the bcc inclusions or the influence of hard inclusions on the soft matrix. Third phenomenon is related to evolution of the mechanical and radiation induced damage fields, described in the framework of Continuum Damage Mechanics. In order to develop the constitutive models, the materials have to be characterized by means of cryogenic testing and microstructural observations (Fig. 1). Unique set-up for complex loads consisting of traction and torsion, allowing to perform the non-proportional loading paths, is used. The instrumentation involves the piezoelectric load cell, pair of clip-on extensometers, set of special thermistors for extremely low temperatures and dedicated set of tensometers.



Figure 1: Characterization of modern Cu-Nb<sub>3</sub>Sn low temperature superconductors

Acknowledgments: grant of National Science Centre 2021/41/B/ST8/01284 is acknowledged

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## Properties of AM versus classical materials at extremely low temperatures

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Novel production techniques, consisting in additive manufacturing (AM), allow to design the physical and mechanical properties of the manufactured components. Such components may operate at extremely low temperatures, provided their properties are suitable for extreme cryogenic conditions. Among typical low temperature applications are the cryostat components, the thermal shields, the cryogenic transfer lines as well as the superconducting magnets. It is therefore of primary importance to characterize the behavior of AM materials near absolute zero, when compared to the classical materials manufactured by using standard methods (casting, forging, etc.). In particular, it is crucial to analyze the behavior of stainless steels, massively used at very low temperatures, in the form obtained by means of AM and determine their inelastic response under monotonic or cyclic loads.

One of the most interesting questions, arising during the experimental investigations, consists in elucidating the physical phenomena observed in the AM stainless steels strained in cryogenic conditions. Given their different microstructure, resulting from AM, does the intermittent (serrated) plastic flow occur? Does the plastic strain induced fcc-bcc phase transformation take place [1]? What is the susceptibility of the AM stainless steel to microdamage evolution? All these questions are addressed by applying the methods of experimental mechanics. In particular, the experiments performed on the AM stainless steel specimens are compared with the experiments performed by means of the classically manufactured samples (Fig. 1). Moreover, the conditions of fracture as well as the fracture induced microstructure evolution in both types of stainless steel are discussed.



Figure 1: Fracture: 316L AM vs. standard make; phase transformation in standard 316L at 4.2 K

Acknowledgments: grant of National Science Centre 2021/41/B/ST8/01284 is acknowledged

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#### Full-field optical metrology for testing and monitoring of micro and macro 3D engineering structures

L1: Coding information with optical methods L2: Interferometric methods and their applications L3: Incoherent light methods and their applications

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Optical methods provide means to measure an arbitrary 3D technical and biological object in fieldwise and noncontact manner in wide range from  $\mu$ m up to m) and with high sensitivity (from nm to mm) (Fig.1a). All of them capture information about complex amplitude of an object in the form of intensity images (most often fringe patterns) followed by numerical decoding of phase information and converting it into cloud of points (x,y,z) or 3D displacements and strains (Fig.1b).



Figure 1: Advantages of full-field optical metrology a) and its applications b).

The lectures provide at first optonumerical background for the methods of phase retrieval and discus the rules for the proper choice of a measurement system based on coherent or incoherent light. Next, numerous examples of applications (for macro, meso and micro scale objects) of interferometric, speckle, holographic, shearography, moire, fringe projection and digital image correlation methods are provided with focus on solving a variety of experimental mechanics and civil engineering problems. Finally the examples of hierarchical and multimodal measurement systems are presented and their role in creating digital twins of a mechanical or biological structures is discussed.

Acknowledgments: The Polish Metrology Program, grants PM/SP/0079/2021/1, PM/SP/0058/2021/1

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#### Pressure Stimulated Currents versus Acoustic Emissions in laboratory and structural experiments

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The most mature and widely used technique for Structural Health Monitoring (SHM) is that of the Acoustic Emissions (AE) [1]. In parallel, it is experimentally verified that during mechanical loading of specific classes of materials, damage mechanisms are activated, generating electromagnetic emissions [2], a specific type of which, the weak electric currents, denoted as Pressure Stimulated Currents [3], is considered a promising tool for SHM. The underlying mechanisms generating PSCs are related to the genesis of micro-cracks, which are the origin of motion of electric charges and consequently of changes of local polarizations.

In this study, the acoustic and electric activities, detected while concrete and rocklike specimens are loaded mechanically, are considered in parallel, in order to comparatively assess their outcomes and detect potential characteristics that can be considered as prefailure indicators. The study revealed satisfactory agreement between the evolution of parameters quantifying the acoustic activity and the respective evolution of the PSC, both for elementary and structural tests (Fig.1). It was highlighted that the combined used of the two techniques offers better insight into the damage mechanisms activated, permitting safer quantification of the remaining life (and load carrying capacity) of the member studied.



**Figure 1**: The temporal variation of the PSC vs. that of the strain developed during uniaxial compression of brittle materials (left), and the acoustic activity (in terms of the F-function) vs. the electric one (in terms of the PSC) (right). An excellent agreement between them can be seen.

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#### Detection of criticality conditions using Acoustic Emissions and concepts of "Non-Extensive Statistical Mechanics"

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A novel procedure is discussed, aiming to improve the exploitation of data provided by the Acoustic Emissions (AE) technique, in the direction of detecting potential pre-failure indicators. The approach is based on "Non-Extensive Statistical Mechanics" (NESM), a discipline founded on the concept of non-additive entropies  $S_q$  [1]. The underlying feature of these entropies is that they violate the additivity principle (the entropy of a system is not equal to the sum of the entropies of the individual parts constituting the system), and it is expressed in terms of the entropic index, q, a measure of the system's non-extensivity, accounting for non-independent, long-range interacting subsystems with memory effects.

In order to highlight the potentials of this approach, the cumulative distribution of the interevent time intervals between successive acoustic hits is considered, as obtained from long series of experimental protocols. The analysis revealed that the temporal evolution of the entropic index q (strongly depending on the homogeneity degree of the loaded specimen/ structure) provides useful indices (Fig.1), warning about entrance of the loaded system into its critical stage (namely, that of impending fracture), in agreement with indices provided by the evolution of alternative AE parameters like the F-function [2, 3], the Ib-value, etc.



**Figure 1**: The temporal variation of the entropic index q vs. that of the load, for a 3P-bending test of a plain concrete beam: q is maximized well before the load attains its own maximum value, providing a clearly distinguishable index about entrance of the beam into the stage of impending fracture.

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## Exploring the acoustic activity in mechanically loaded specimens and structural elements in the "Natural Time Domain"

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In this study the acoustic activity, generated while structures or specimens are loaded mechanically, is explored by means of the "Natural Time" (NT) [1]. NT was introduced to study seismic phenomena, and gradually it was proven advantageous for the description of a variety of complex dynamic systems, since it allows optimal extraction of signal information from systems approaching criticality. The key aspect of the analysis by means of NT is that only the sequence (order) of the events is kept (together with their energy) ignoring the conventional time at which an event occurs. Hence, NT is in sharp contrast with the hitherto used conventional time t, modeled as the one-dimensional continuum R of the real numbers. In such a way, NT analysis allows one to study how the energy of the events varies while the system progresses to a possibly new state that may lead to an extreme event.

Applying the NT analysis for time series of acoustic events gathered from a variety of experimental protocols (both elementary and structural) employing the F-function [2], it was proven that its values increase almost imperceptibly for the major portion of the loading process. On the contrary, during the last loading stages, the F-function increases abruptly, governed systematically by a power law of the form  $F(\chi)=A\chi^m$  (Fig.1). The onset of validity of this law is proven to be an early signal warning about the entrance of the loaded system (specimen) into the stage of impending macroscopic fracture.



**Figure 1**: The temporal evolution of the load applied on a cement-mortar beam under 3P-bending in terms of the conventional time (vs. that of the amplitude of the AEs) (left) and the respective evolution in the NT domain (vs. that of the F-function) (right). The portion of  $F(\chi)$  governed by the  $F(\chi)=A\chi^m$  law (the onset of which designates entrance to criticality) is marked red.

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#### Maximizing energy efficiency of structures manufactured using additive techniques - a review study

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Like other metamaterials, cellular structures have programmable properties. The mechanical properties of cellular structures include high stiffness and strength per unit mass; thus, these materials are commonly used in a wide range of engineering applications in the automotive, aerospace, and aeronautic industries. Because cellular structures undergo controlled deformation, they have great potential in energy absorption applications.

The increasing popularity and accessibility of additive manufacturing techniques have sparked a surge in interest in regular cellular structures as effective mechanical energy absorbers. The versatility of 3D printing allows for creating intricate structures with relative ease and speed, using a broad spectrum of materials, including plastics and metallic alloys [1]. This has led to a wealth of research publications exploring the capabilities and applications of these structures [2,3].

The main objective of the present research is to explore the potential fields of application for the energy-absorption structures. Selected studies consisting of experiments as well as numerical simulations of several types of various 3D-printed topologies are broadly discussed. In the first part of the paper, the main focus is applied on additive manufacturing problems and experimental research within various loading conditions. The experimental tests of cellular topologies are presented and material characterization studies for constitutive modelling is also discussed (Figure 1).



Figure 1: (a) Honeycomb topology manufactured using LENS technology, (b) lattice topology tested using DSHPB setup





In the second part of the paper, the main focus is applied on modelling and simulations problems connected with additive manufacturing and cellular topologies testing (Figure 2). Various modelling approaches are presented and different types of materials and topologies are discussed, which were tested within various loading conditions. Furthermore, importance of a proper constitutive modelling is highlighted with the selected examples to feasible reproduce failure and deformation of the given materials.



**Figure 2**: Numerical simulations of: (a) star-triangular topology made of ABS-plus [4], (b) Honeycomb made of titanium alloy [5]

Acknowledgments: The research was supported by the National Science Centre under research grant No. 2015/17/B/ST8/00825.

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## **IPPT INVITED LECTURES**





## On the Non-standard Experimental Methods for Mechanical Characterization of Conventional and Printed Materials

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The lecture is devoted to three non-standard experimental techniques used for complex characterization of a wide range of materials. Among them one can indicate: yield surface concept; testing of thin sheets under compression within a large range of deformation; dynamic tests for a wide range of strain rates.

In practice, many of engineering materials are not isotropic. In such cases investigation of a yield surface is regarded as one of the most effective methods to study anisotropic properties of materials. Yield loci can be represented in a stress space by the experimental points determined on the basis of stress-strain diagrams for the magnitude of the effective strain assumed as a yield definition. Details of this technique will be discussed based on the experimental data captured for selected materials.

The second non-standard testing method presented in this lecture deals with the mechanical properties characterization of thin sheets. An application of compressive loading usually leads to specimen buckling. To avoid this phenomenon, specialised fixtures are used to support specimen during the test. There are many designs of fixture suitable for monotonic compression. The prototype of an innovative fixture suitable not only for monotonic compression, but also for tension–compression cycles was designed and manufactured by the IPPT research workers. It enables to avoid buckling during compression of specimens made of thin metal sheet. The friction force, which is generated because of a movement of two parts of the fixture, is measured by the special strain gauge system during each test. It allows the elimination of friction force influence on the stress–strain characteristics.

The last type of experimental non-standard techniques presented in this lecture will be devoted to identification of dynamic properties of materials using different kinds of the Split Hopkinson Pressure Bars.

Acknowledgments: The work supported by the National Science Centre through the Grant No 2019/35/B/ST8/03151

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## Additive Manufacturing as a new opportunity for lightweight Shape Memory Polymers in industrial applications such as robotics or medical surgery

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Shape memory polymers (SMPs) are multifunctional materials that can change their shape under external stimuli, usually temperature, gaining interest in various applications [1, 2]. Particularly interesting is a new generation of multiple SMPs that demonstrate the ability to memorize more than two shapes. The property significantly broadens the functionality of SMPs making them attractive for applications from robotics to biomedicine. The multiple shape memory effect (SME) can be achieved through additive manufacturing (AM), particularly 4D printing. The approach facilitates the design and development of devices with complex structures unattainable by traditional techniques [3].

Thermoset shape memory photopolymer specimens are printed by laserstereolithography and digital light processing. The investigation of their thermal, mechanical and thermomechanical properties by Discovery TGA 5500, Discovery DSC 250 and Instron 5969 testing machine with thermal chamber is conducted.

Complex-shape SMP actuators with multiple SMEs will be designed, processed by AM technologies and optimized by prototyping facilities and computational modeling (CAD, FEM, topology optimization software). The actuation to trigger the shape change of multiple SMPs is obtained by step-by-step heating to various temperatures.

Lightweight shape-morphing devices for various applications, including minimally invasive surgical devices and smart robotic grippers will be developed and prototyped.

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## Multiscale modelling of thermomechanical properties of advanced materials

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Multiscale modelling is probably one of the most intensively developing modelling methodology in the scope of mechanics and material science. The goal of such research is to estimate the macroscopic averaged properties of heterogeneous material knowing the properties of the phases at the micro-level and morphological features and space distribution of components within the material volume. In view of their microstructure the heterogeneous media can be subdivided into those with periodic or random microstructure. In the first case a unit cell can be defined which by its multiplication in three directions fills the material volume. Materials with this type of microstructure are mainly synthetic ones like e.g. meta- materials produced by additive manufacturing. In the second case the microstructure can be characterized by a set of statistical distribution functions of same microstructural parameters: size and shape of components or their spatial distributions (packing) in the material volume. Among such materials there are natural (e.g. metal polycrystals) and synthetic (e.g. composites) materials.

The key component of the individual micromechanical models is a micro-macro transition scheme that enables to estimate the sensitivity of the macroscopic response to the changes in material microstructure and local properties. There are two basic methods used within multiscale modelling: (i) mean-field methods which are based on the analytical solutions obtained in the frame of linear thermoelasticity or thermal conductivity for a unit cell, applicable usually to particulate microstructures [1,2] or polycrystalline metals [3], and (ii) full-field numerical homogenizations which enables consideration of more complicated morphologies [4]. Fundamentals of both methods will be discussed within the lecture.

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# Additive manufacturing of metal matrix – particle reinforced composites

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Additive manufacturing, commonly referred as 3D printing, has become very popular in recent years, both in industry and academia. The basis for such wide-spread interest is undeniable advantage of fast prototyping, even for complex structures, also for advanced materials. Materials as well as fabrication approaches can be used for classification of additive manufacturing. We can produce polymers (plastics), metals, and ceramics. Also, composites (mixtures of different materials) are getting attention. Usually, this approach leads to complex problems as different materials tend to behave differently when exposed to physical and chemical processes.

As an example, a typical process of metal matrix composite manufacturing can be described as below: (1) mixing metallic and ceramic powders (2) spreading of one layer of such mixture (3) applying a lot of heat to powders by a scanning probe. As a results of scanning (3) the powders get more plastic and even melt, and eventually connect. This happens only where the scanning applied heat. A laser is usually a heat source. When the first layer of powders is done scanning, the second layer is added. This way the whole bulk of planned geometry is fabricated with appropriate scanning strategy.

Such an approach creates several problems. Firstly, in step (1) the mixture composition is fixed. If we want to change it, we need to prepare a new mixture. Secondly, in step (2) the same mixture is used so the whole part is being built from the same material. As can be expected, it is not always essential and is usually economically unjustified, especially for expensive materials. Finally, the ceramic powder, usually nanopowder, may significantly agglomerate and reduce the mechanical strength of the metal-ceramic interface and the overall mechanical properties of the printed composite.

During this presentation we will discuss the impact of above mentioned problems and the solution for them, which basically offer conducting steps (1) and (2) for just one powder (cheaper) and eventually adding the second powder only in the spots where it is really needed from technical viewpoint. As an example where such an approach would be beneficial, we can discuss friction pair. In typical tribological pair, the wear occur only at small surface compared to the whole bulk. It means that reinforced mechanical properties are needed in relatively small fraction of part's volume. With usual additive manufacturing strategy, the whole volume would be made of the composite. We will discuss how to manufacture parts, where reinforcement is added only in prescribed spots of parts, i.e. where actual wear is expected.

Finally, the initial results of engineering work on 3D printer with a special distribution module will be presented.

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#### Biomedical shape memory alloys versus experimental mechanics and additive manufacturing

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Recently, much effort has been made to develop biomedical Ti-based shape memory alloys (SMAs) in order to overcome certain drawbacks of the alloys commonly used for implant applications i.e. high Young modulus of commercially pure Ti or Ti–6Al–4V alloy (greater than 100 GPa) when compared to that of the human bone (10–30 GPa), poor malleability of Ti-Ni based SMAs and allergenic content of selected elements e.g. Ni. Numerous metastable  $\beta$ -Ti alloy systems have been explored using mainly conventional manufacturing techniques [1]. Superior mechanical characteristics including ultralow Young modulus of 40 GPa, nonlinear, tuneable superelasticity, near-zero hysteresis and good plasticity have been obtained. These properties originate in particular from specific crystallographic textures and unconventional deformation mechanisms e.g. high-order-like (continuous) strain glass transition associated with nanosized modulated domains (nanodomains) caused by randomly distributed interstitial atoms of oxygen or nitrogen [2].

This talk will deal with the latest advancements in the field of the biomedical mainly, but not exclusively, Ti-based SMAs. Specific approaches for investigation of these alloys during deformation by means of selected in-situ techniques of experimental mechanics including among others digital image correlation, infrared thermography and acoustic emission will be presented [3, 4]. Results of the microstructural, kinematic, thermal and acoustic aspects of the activity of the underlying phase transformations of the new SMAs under mechanical or thermal loadings will be discussed. Furthermore, challenges and opportunities concerning additive manufacturing with a special focus placed on the fabrication of the biomedical SMAs for implant applications will be reviewed.

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### YOUNG RESEARCHER SESSION





#### A Study on Subystems for an Unmanned Underwater Vehicle

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This study proposes a selection of structure systems and subsystems for an autonomous underwater vehicle (UUV) capable of operating in maritime environments where operation by humans is unsafe due to geographic characteristics or the type of mission. This is a study integrated into an underwater vehicle construction project where, in addition to GPS navigation system, astronomical navigation is used for maneuvering and geo-location. [1] [2] The project aims to improve the capacity for underwater exploration, surveillance and monitoring of mines and other equipment that put a Nation at risk. To consolidate the research, several bibliographic sources were consulted, including scientific works and articles. Specific requirements were derived from analysis of mission needs and characteristics of anticipated operating environments [3] [4]. This study contributes to the advancement of UUV technology in the ongoing project.



Figure 1: Methodology for define sub-systems of UUV





The structural design of the UUV is a crucial point for future operations, aiming to ensure resistance to underwater pressure, maneuverability, and payload capacity. It is recommended to adopt lightweight and resistant composite materials, combined with advanced manufacturing techniques, as suggested by some authors [1] [2]. For that we intent to use boards in a first phase and a 3D printer to concept the installation of the subsystems and propulsion systems. To define the sub-system it is intended to use the methodology in figure nr1.

Therefore, the subsystems to be considered when designing the technical specifications are the following [5] [6]: structural design; the material of the main structure and support structure; power system; propulsion system; ballast system?; communication system; navigation system.

In summary, this study pretends to offer a comprehensive view of the subsystems to integrate in a UUV. Next work will be defining the technical specifications of the chosen subsystems to build a UUV with high performance and resistance in operation. This work is expected to contribute significantly to the development of UUV technology.

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#### An evolution of yield surface for Ti-Cu bimetal after plastic predeformation under complex loading

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Alloys were developed to overcome the limitations of pure metals, achieved through conventional alloying, mixing metals or non-metals in powder or molten states. They offer enhanced properties like toughness, strength and corrosion resistance. Over centuries, alloys like stainless steels, Cu-based alloys, Ni-based super alloys (Inconel) and Ti-based alloys have been developed for diverse industrial uses. Whereas, bimetallic structures, combining two different metals, can overcome individual material limitations while maintaining desired properties [1]. This approach addresses material selection challenges and allows customization based on specific performance requirements in engineering applications. The selection of manufacturing techniques and component materials substantially impacts the mechanical properties of bimetals. Therefore, this research presents an experimental and theoretical investigation by applying the yield surface approach to identify the physical mechanisms responsible for the plastic deformation of Ti-Cu bimetal resulting from the complex mechanical loading.

The initial yield surface of Ti-Cu bimetal and its evolution were determined by the sequential probe technique using single specimen along 17 different strain-controlled directions in the plane stress state [2]. The subsequent yield surfaces were determined after plastic pre-deformation caused by monotonic tension and combined monotonic tension-cyclic torsion up to 1% axial strain.

It was found that the initial yield surface obtained for the as-received Ti-Cu bimetal exhibits anisotropic behaviour. Such behaviour could have come from the preferred texture obtained during the bimetal production. The yield surface after monotonic tensile predeformation shows a significant shift towards the tensile direction, i.e. kinematic hardening within the bimetal. Whereas, pre-deformation caused by combined monotonic tension-cyclic torsion leads to the significant softening of the bimetal components in all directions.

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#### Microstructural analysis of austenitic stainless steels during monotonic and cyclic loading at extremely low temperatures

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The stainless steels are massively applied at extremely low temperatures in view of their excellent properties, including ductility. However, they behave in a metastable way when strained beyond the yield point. The aim of the present work is to observe and analyse the evolution of the material microstructure, in particular the fcc-bcc phase transformation, during the plastic flow and fracture in grades AISI304 and 316L stainless steels at extremely low temperatures (77K and 4.2K). In addition, the extended multiscale constitutive model, describing the phase transformation during cyclic loading, will be presented.

The unique cryogenic experiments were performed by using an in-house test system. The specimens were mounted in the vacuum insulated cryostat, located in the dedicated testing machine. In order to ensure suitable temperature, the coolant was conveyed from dewar to the cryostat via special transfer line. The fracture area was studied in detail by using the magnetometric methods, the scanning electron microscopy (SEM), the electron backscatter diffraction (EBSD) technique as well as the X-ray diffraction by means of the synchrotron radiation. The X-ray diffraction studies revealed – among others - that the fccbcc phase transformation accompanies massively the macrocrack propagation [1]. Moreover, the resulting  $\alpha$ ' martensite intensively twins to achieve good lattice fit to the austenitic matrix. The secondary phase nucleates at the intersections of the shear bands, i.e. at the crossing of  $\varepsilon$  martensite plates or twin lamella, then undergoes growth and coalescence [2]. Additional studies were carried out to explain the microstructure evolution in the austenitic stainless steels subjected to cyclic loads at near 0 K temperatures. Rapid increase of the secondary phase was noticed, resulting from the phase transformation during the subsequent loading cycles. Conditions of accelerated shakedown and of ratchetting were investigated. Also, the analysis of fracture in the context of cyclic loads, including the microstructure evolution, was performed.

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#### Analysis of the mechanical behavior of I-members built from adhesive-bonded cold-formed steel channels

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The evolution of structural adhesives has significantly expanded their application within the engineering industry, ranging from structural connections to retrofitting existing structures [1]. The concept of fabricating structural components through adhesive bonding represents a pivotal innovation [2]. Utilizing adhesives offers several advantages over traditional mechanical connectors, such as improved fatigue resistance, accelerated construction processes, uniform stress distribution, and enhanced quality assurance. Despite these benefits, adhesive-bonded joints and structures have limitations due to their susceptibility to geometric and material imperfections, restricting their widespread use [3].

The proposed research is a comprehensive study, encompassing empirical, numerical and theoretical approaches. It aims to analyze the mechanical behavior of steel thin-walled I-members, consisting of two cold-formed channels permanently bonded together by webs with a structural adhesive (Fig. 1). The project will start with the experimental investigation, followed by the formulation of numerical and theoretical mathematical models for the I-members. These models will assess the response of the members to compression and bending loads within a geometrically and physically nonlinear range.



Figure 1: Schematic diagram of the member (a); concept diagrams of web ribbings (b)

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## Development of assessment tools for bolted joints using AI-driven optimization techniques

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Bolted joints are widely used in the aerospace and automotive industries due to their ease of assembly, disassembly and design flexibility. Optimizing these joints is essential to achieve uniform load distribution and minimize the number of bolts required, thereby reducing system cost and weight. Bolted joints can be optimized based on bolt's configuration/layout, stresses on the threads, size of bolt, tightening sequences, and tightening strategies [1]. This research aims the development of assessment tools for bolted joints to ease the bolt's selection process, specifically targeting the optimization of bolted joint stiffness. The final product might be a software or an automation tool that inputs variables and outputs optimized solutions.

Among various optimization techniques, those based on evolutionary algorithms are preferred for their robustness and ability to handle multiple variables [2]. Genetic algorithms, known for their effectiveness in finding global optimal solutions, are widely applied in engineering optimization problems [3]. Therefore, genetic algorithms have been selected to optimize decision variables in order to maximize or minimize the objective function. Moreover, Python a programming language has been used to implement these algorithms. There is a possibility in integrating Python with ANSYS (FE package) to enhance the effectiveness of the tool. However, using finite element (FE) solutions can be both time-consuming and computationally expensive. The research seeks a solution that is computationally efficient and fast.

Initially, the focus is on optimizing the bolt's layout and size under various loading conditions. This model will later be validated using more finite element analysis software or, if feasible, experimental methods. The initial geometry for bolt placement, with constraints, is illustrated in Figure 1. It shows four bolts positioned at the corners of a plate of cross-section 250x250 mm. A central beam, depicted in red, is attached to the plate with bolts and is subject to a specific moment. The search space for bolt placement is limited to the area between the dark and red regions, due to geometric constraints.



Figure 1. Four bolt model with a beam at the centre of the plate under moment

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#### Autonomous Robotic Tribology Testing System for Lubricated Hot Aluminum Blanks

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This study reports an autonomous experimental setup designed to perform multicycle tribological characterization on heated metallic blanks by integrating a UR10 robotic arm. The system incorporates a custom-designed pin holder mounted on the robotic arm to securely hold the die material pin [1], enabling tribological evaluation across a wide range of applied normal loads and sliding velocities against the heated blank strip. Prior to testing, liquid lubricants are uniformly applied onto the heated blanks utilizing an automated spray gun apparatus. The robotic manipulator is seamlessly integrated with the Robot Operating System (ROS) framework [2], facilitating autonomous control of the arm kinematics and synchronized data acquisition via the UR ROS Driver interface [3].

Upon completion of each tribological cycle, the friction data is automatically recorded, processed, and visualized through the implementation of advanced algorithmic routines developed in Python, significantly reducing the requirement for manual data handling by the researcher. Results are presented elucidating the coefficients of friction (COF) obtained during sliding tests performed on 2 mm thick aluminum alloy AA6111 blanks at elevated temperatures of 300°C and 350°C. The autonomous nature of this experimental setup streamlines the overall workflow, promoting systematic and efficient data collection, processing, and characterization procedures. Highlighting the benefits of robotic process automation and integrated software paradigms within the domain of tribological research, this system notably enhances experimental throughput. The study comprehensively details the system architecture, experimental methodology, and showcases salient results acquired from the autonomous tribological evaluation of lubricated hot aluminum blanks.

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#### **Carbon coatings: DLC films**

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This research presents the effect of the reaction atmosphere and the chemical composition of steel on the production of carbon coatings. The aim of the project under which the following studies have been carried out is to create a wear-resistant coatings dedicated to gear teeth for conveyor drive units operating in harsh environments. Radio frequency chemical vapour deposition (RFCVD) processes were conducted on a substrate of 35CrSiMn5-5-4 steel (35HGS) and comparatively on 66SiMnCrMo6-6-4 steel (OVAKO677L). A total of 8 processes were performed in which the composition of the reaction atmosphere was altered. Thin DLC films have been produced, which are characterized by the presence of three distinctive bonds, specifically sp<sup>3</sup> (figure 1) [1]. Examination of how the ratio of methane to nitrogen and hydrogen affects selected properties of DLC films was performed. The adhesion of the coating to the surface was analyzed. Values of hardness and reduced Young's modulus were obtained through nanoindentation tests.



Figure 1: The sp<sup>3</sup>, sp<sup>2</sup>, sp<sup>1</sup> hybridised bonding [1]

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# Porous silicone networks as potential scaffolds in tissue engineering – mechanical aspects

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Porous flexible structures with good biocompatibility between the scaffolds architecture as well as the biological environment brought to, have been subjects of interest for many years but are still proven challenging to be achieved.

Synthetic polymers such as silicones have been widely used in medical devices since they possess multiple advantages, as they present biocompatibility and can be produced in large quantities, being easy to process at low costs. Besides all of these, the structure– property relationship of the precursor polymers has been extensively studied and is very well-established. Nowadays porous silicone networks can be generated through multiple, optimized techniques that require fast obtaining time with no catalysts involved and minimal to none solvents [1].

It is also well known that mechanical properties, such as elongation at break and compressibility can provide an important insight into the ability of the materials to withstand different conditions. These structures must be mechanically stable *in vivo*, and based on the location and tissue type (hard or soft), have to provide good load-bearing properties, demand that silicone materials fulfill well. The mechanical tests of the silicone porous networks indicated good compressive strain results (up to 50 %) after the immersion in the selected biological medium.



Figure 1: Compressive strain of porous silicone network samples after immersion in biological medium

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# Cryogels based on poly(vinyl alcohol) and a copolymacrolactone system

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Polymer blending is the most useful method for improving or modifying the physicochemical properties of the material [1]. In this study, we introduce a new cryogel system based on poly(vinyl alcohol) (PVA) and poly(ethylene brassylate–co–squaric acid) (PEBSA). The chemical composition, thermal stability, viscoelastic properties, and network morphology were investigated by Fourier Transform Infrared Spectroscopy (FT–IR), Thermogravimetric Analysis (TGA and DTG), Dynamic Mechanical Analysis (DMA), and Scanning Electron Microscopy (SEM).

The viscoelastic characteristics (E', E'' and tan  $\delta$ ) are constant during the investigated frequency range, however with higher E' values for PVA–PEBSA (50/50). The study highlighted improvements in the flexibility of the matrix network due to the intermolecular chain interactions brought by the introduction of PEBSA in the cryogel structure. The new system will be used for further incorporation of hydrophobic bioactive agents for various applications.



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#### Mesostructure modification of additively manufactured lattice materials towards improved mechanical properties

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The mechanical properties of metamaterials obtained with additive manufacturing strongly depend on the designed geometry of the mesostructure. Such materials are used, for example, in medical implants, heat exchangers and energy absorbers. The basic parameters of a single-phase material's mesostructure include the unit cell's size and shape, the thickness of walls and struts, and the resulting porosity and pore size. The properties of the base material are also important. The range of mesostructure parameters largely depends on the manufacturing technique, which in the case of metals is Laser Powder Bed Fusion (L-PBF), also known as Selective Laser Melting (SLM).

Due to the high production cost and specialised applications, maximum durability of the material is desired while maintaining the operational and strength parameters. It is required to reduce stress and strain gradients in the structure. The modelling aims to minimise stress concentration by eliminating notches in the designed mesostructure geometry [1]. The study examined the influence of the curvature of internal surfaces on the mechanical properties of the metamaterial. Several sets of geometrical models of structures with various parameters were designed in the Siemens NX program. Geometrically and materially nonlinear analyses (GMNA) were performed using the SIMULIA Abaqus software. The structural strength was assessed at the macroscopic and mesoscopic levels, adopting various yield criteria. The material model was determined experimentally and numerically for the Ti6Al4V base material as a true equivalent stress-strain relationship [2].

Based on the simulation results, it was found that modifying the radii of curvature of the mesostructure allows for a significant increase in the effective stiffness and strength of the metamaterial. The results of numerical calculations constitute the basis for conducting experimental tests of the material's mechanical properties on samples made using SLM technology.

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# Resistivity variation of carbon-based nanomaterials in lime-based restoration pastes

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The present article investigates the dispersion of nanomaterials in ternary lime-based cementitious nanocomposites for applications related to the restoration of Cultural Heritage Greek Monuments of the 19<sup>th</sup>-20<sup>th</sup> century (Fig.1i), in laboratory scale. The working principle aims at transforming the matrix into a sensor by dispersing carbon-based materials (CBNs) to form conductive paths. Different ratios of superplasticizer (SP) per (CBNs) were examined, at a content equal to 0.15 % of the binder. Monotonic compression and compressive cyclic loading up to 50% of the corresponding ultimate strength with simultaneous application of Direct Current (DC) were conducted. The fractional change of the electrical resistance that was measured under cyclic loading-unloading loops (at a level of 20 % of the compressive strength) was equal to 12% (Fig. 1ii). All the pastes exhibited fully-recoverable electrical resistances at the end of each cycle, in accordance with similar findings by Dalla et al. (2020) [1] (for the same percentage of the peak compressive strength, which varied in an inverse relation with respect to the applied stress due to the instant compaction level).



**Figure 1**: i) Demonstrator on a greek Island ii) Piezoresistive response of the prismatic specimen made of: 0.15 wt% nano-reinforced paste at maximum load at 20% of the compressive strength

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#### The method for residual strains determination in WAAM samples

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The method for non-destructive determination of residual strains ( $\varepsilon_{res}$ ) distribution in the stainless-steel samples produced by the wire arc additive manufacturing (WAAM) is presented. The method relies on full-field monitoring of displacements and strains during step-wise releasing of residual stresses from the sample manufactured at the pre-clamped substrate. Monitoring is performed by means of 3D digital image correlation method (3D DIC). 3D DIC system, the example of clamped sample and the results after partial realising of  $\varepsilon_{res}$  are presented in Figure 1. The samples produced by WAAM using different technological parameters (current, voltage, cooling process) and sample parameters (wall height, material) are investigated. The results are analysed and compared, in order to support FEM modelling of residual stresses distribution and optimization of WAAM parameters (including the novel in-process cooling system) from the point of view of minimizing residual stresses.



**Figure 1**: (a) The 3D DIC measurement setup for residual strain measurements; (b) the absolute displacement map of the sample; (c) the absolute displacement plot for the A-A cross-section.

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#### Fatigue Limit determination using Static Thermographic Method

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The subject of the research is the determination of the fatigue limit of reinforced polymers using the static thermographic method. The method was proposed by Risitano and Risitano [1] and uses the measurement of surface temperature during a static tensile testing. The investigated material was PC GF10. The experimental investigation was performed using a tensile machine Zwick/Roell Z005 and the surface temperature was monitored with a thermographic camera. The camera used for monitoring is a FlirA40. The thermographic measurements were taken at a framerate of 50 Hz on the fracture area. From tensile tests, the stress over time was exported and overlapped with the temperature over time to determine the fatigue limit of the material. A thermographic map is presented in figure 1. Figure 2 represents the setup for the tensile tests with the thermographic camera. The investigated material was PC GF10. The values of the fatigue limit were compared with the fatigue curves of the material [2]. The fatigue limit is present between the first two phases of temperature. The first phase represents a linear decrease due to thermoelastic effect, followed by a deviation from linearity until a minimum point (second phase) [3].

A first conclusion of this investigation is that the fatigue limit can be determined using the static thermographic method (STM), observing the change between the two phases of the surface temperature.





Figure 1: Thermographic measurement

Figure 2: Test setup for measurements

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### Microstructure, mechanical and tribological properties of (TiTaNbZr)<sub>100-x</sub>Cu<sub>x</sub> high entropy alloys for potential antibacterial properties

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The  $\beta$ -Ti-based alloys are widely applied in medicine. However, the strong influence of biomedical high entropy alloys (bio-HEAs) and antibacterial high entropy alloys (AHEAs) was underlined recently. The chemical compositions of bio-HEAs are mainly composed of Ti, Ta, Nb, Zr, Mo and Hf. The investigations confirmed high biocompatibility and promising mechanical properties [1]. The newest literature data also described the strong demand for combined biomedical and antibacterial properties of produced high entropy materials. The combination of both properties could be reached by the addition of antibacterial chemical elements such as copper (Cu) [2]. It was revealed that *Escherichia coli, Staphylococcus aureus*, or *Klebsiella aerogenes* could be classified as copper ions (Cu<sup>2+</sup>) sensitive bacteria [2].

In the presented studies, the vacuum arc melted and thermally treated  $(TiTaNbZr)_{100-x}Cu_x$  (where: x = 0, 10 and 20 at. %) high entropy alloys will be discussed. Experimental results confirmed multi-phase structure, segregations of the alloying elements in the microstructure, an increase of microhardness, reduced Young's modulus and improved wear resistance with increased Cu-content. A high level of biocompatibility was also confirmed.



Figure 1: Osteoblast cell morphology on studied Cu-containing HEAs

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#### Vibration-Based Piezoelectric Energy Harvesting – A review

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The research topic focuses on the mathematical modeling of structures that harvest energy from vibrations [1]. The energy crisis, global warming, and environmental pollution have been prominent topics in recent years, driving the search for alternative fuel sources. Renewable energy sources, particularly those that are sustainable and environmentally friendly, are increasingly being explored.

In recent years, researchers have been investigating technologies for harvesting energy from various sources. Among these, kinetic energy, in the form of vibrations and random movements, is abundant in our environment.

Devices that harvest kinetic energy operate based on principles such as piezoelectricity, electromagnetism and electrostatics. Numerous articles have discussed the advantages and disadvantages of each type, with particular attention given to piezoelectricity due to its simplicity, high energy density, little risk to natural environment.

Such devices have gained popularity as alternatives for powering small devices, like wireless sensors, transmitters, actuators, medical implants, etc. The electricity source is the mechanical work performed by the piezoelectric material while being deformed, eliminating the need for external electrical sources or battery replacements in some cases.

One of the most commonly used piezoelectric materials is PZT which is an inorganic ceramic material synthesized in laboratories. It is known for its high strength and relatively low cost.

The most popular method of energy harvesting using piezoelectric materials is through vibrating structures [2], with cantilever beams being the most prevalent [3]. These devices operate in mode 31, where the beam is designed to bend, resulting in the generation of alternating electric current.

The amplitude of vibration is crucial for the amount of charge generated, depending on the frequency of the external stimulus. However, the frequencies at which the devices perform best are limited to a narrow range, posing a challenge that researchers seek to overcome through various means.

The primary goal of the research is to conduct a thorough investigation of a device that harvests vibrational energy. This includes designing a conceptual structure, developing a mathematical model to describe the relationship between mechanical deformation and generated current, conducting experiments to validate the models and optimizing the solution for the device's most effective operation.

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#### Innovations in Thermal Barrier Coatings: Exploring Alternative Materials and Approaches

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Thermal barrier coatings (TBC) play a pivotal role in protecting high-temperature components in aerospace, power generation, and automotive industries. These coatings, which are applied onto the surface of the substrate provide thermal insulation and resistance to chemical degradation. Currently, Yttria-stabilized zirconia (YSZ) is the most widely used TBC material, owing to its high-temperature stability, low thermal conductivity, and hightemperature resistance to chemical corrosion. However, as the operating temperature increased, YSZ receded from its performance, ultimately steering the focus of research towards alternative coating materials. Some of them include LHA-doped ceria (Ce-LHA) [1] pyrochlores  $Ln_2Zr_2O_7$  (Ln=La to Gd) and fluorite structures  $La_2Ce_2O_7$ . Other approaches include the addition of metal oxides like Re<sub>2</sub>O<sub>3</sub> (Re=La-Gd) [2,3] as dopants to form new compounds, Calcium-magnesium alumino silicate (CMAS) corrosion is a critical factor that causes the failure of TBC. This significantly alters the performance and life of the coating, thus the dynamic nature of temperature and corrosion effects because of CMAS exposure has garnered extensive research recently. Limiting factors of these studies include the accuracy and the probability for the prediction of failure and service life, which experimentally involve complexities and costs that sometimes curb the development of alternative thermal barrier coatings. Henceforth, the use of computational methods [4] to deduce the suitable coating material based on the thermal and mechanical properties could steer the research more effectively and efficiently.

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#### Microstructure impact of additively manufactured and cast Al-Si alloy on the chosen properties of PEO oxide coatings

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Additive manufacturing (AM), commonly known as 3D printing, has revolutionized material forming techniques. One of the leading AM methods is Laser Powder-Bed Fusion, enabling the production of metal parts with enhanced mechanical properties compared to cast alloys. Nonetheless, there remains a necessity to obtain additional functional surface because almost every materials' reaction to the environment begins at the surface.

Plasma Electrolytic Oxidation (PEO) is the most environment-friendly alternative to the conventional electrolytic oxidation processes. However, PEO treatment of Al-Si alloys presents significant challenges [1-3]. The anodization rate of Si particles is notably lower than that of Al phases, resulting in high porosity of the coatings.

In this study, PEO process was conducted on additively manufactured and cast AlSi10Mg alloy. A microstructure and a critical load are discussed. According to the morphology of the substrate (Figure 1), the coatings' phases are better mixed on AM samples. Moreover, these coatings demonstrate better adhesion and scratch resistance.



Figure 1: Examples of microstructure of a) cast b) 3D printed AlSi10Mg alloy

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### Exploring Labyrinthine Sound-Absorbing Composites in Additive Manufacturing: Prototyping, Testing, and Challenges

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Acoustic composites have long been prominent in sound absorption applications. A contemporary trend in acoustics entails exploring complex geometries as matrices and conventional acoustic materials as fillers. Recently, there has been a focus on harnessing the high tortuosity aspects of materials to enhance sound absorption properties [1]. The advent of 3D printing has significantly facilitated rapid prototyping of these materials and validation of their models.

This study focuses on critical aspects of prototyping and experimental testing of acoustic composites fabricated through additive manufacturing. Throughout the material fabrication process, various challenges such as leakages or fabrication errors may arise. These obstacles become evident during experimental validation, where errors can influence the acoustic properties of the material in diverse ways, sometimes yielding unexpected benefits. Throughout the research, commonly used methods for dealing with challenges encountered during experimentation were also verified.

During the process of material investigation, analytical and numerical modelling were conducted, alongside manufacturing and experimental validation of the designed sound-absorbing composite. Experimental trials were carried out employing an impedance tube using standard and unconventional techniques.

In conclusion, this study focuses on prototyping and experimentally testing acoustic composites fabricated through 3D printing. The material fabrication process presents various challenges, which can impact the acoustic properties of the material. The research includes analytical and numerical modelling, as well as experimental validation of the designed sound-absorbing composite using an impedance tube.

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#### In-plane shear investigation of thermoplastic composites at high temperatures using bias extension test

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This study investigates the in-plane shear behavior of thermoplastic composites using a bias extension test. Ensuring a uniform temperature distribution across the sample is critical for successful bias extension tests [1]. To address this, a new fixture was designed and manufactured to preheat the oven before sample placement and minimize the time required for the sample to reach the desired temperature. This also helps to ensure that the sample is not exposed to extreme temperature fluctuations. Several tests were conducted, including thermal tests, and thermo-mechanical bias extension experiments. The Digital Image Correlation (DIC) technique was used to capture local deformations and strains on the specimen's surface during the test. The results show a good agreement between the measurements and the theoretical assumptions of the bias extension test.



Figure 1: Bias extension test setup.

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#### Metal alloys for use as nanoparticle carriers

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In organic reactions, catalysts are employed, which are crucial for enhancing the efficiency and effectiveness of these processes [1]. In an industrial context, where reaction rate and efficiency are paramount, the role of catalysts becomes even more significant. This is particularly pertinent in reactions leading to the formation of products utilized in various industries such as pharmaceutical, cosmetic, or food industries [2]. Currently, in the realm of science and industry, there is a growing demand for increasingly innovative catalysts that are not only durable and efficient but also selective in their action [3]. Much research is focused on seeking new catalytic solutions that can bring benefits such as increased efficiency, reduced production costs, and mitigating negative environmental impact [4]. Numerous catalysts have been described in publications, in which metals play a pivotal role as carriers or catalyst nanoparticles. Studies indicate the diversity of applications of metals such as Fe, Cu, Co, Mo, and Ti in catalysts [5]. Research suggests that each of these metals may exhibit interesting catalytic properties that can be utilized in various chemical processes. The use of these metals as catalysts may be dictated by their ability to activate substrates [5], thermal stability, and other physicochemical properties, such as specific surface area or adsorption capacity [6]. Combining these metals into an alloy can bring benefits, as each component of the alloy can positively influence the reaction, accumulate beneficial effects, and the entire alloy can serve as a carrier for other nanoparticles.

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#### Structure, mechanical properties of amorphous metal alloys system Al<sub>87</sub>(Gd,Y)<sub>5</sub>(Ni,Fe)<sub>8</sub> after short term annealing

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The properties of amorphous metal alloys (AMAs) are determined by the nature of the base metal and the composition of alloying additives [1-2]. One of the urgent research tasks is to optimise the annealing processes of amorphous metal alloy AMA and achieve high values of mechanical properties. The results of X-ray diffraction analysis showed that the nanostructure is formed in the AMA after 5 minutes of annealing and after another 15 minutes it leads to the formation of a microcrystalline structure. The complete replacement of Y by Gd in the Al<sub>87</sub>Gd<sub>5</sub>Ni<sub>4</sub>Fe<sub>4</sub> alloy leads to an increase in the crystallization rate even at T<sub>3</sub>=645±5 K from n=2.46 to n=3.01. The heating time and temperature increase to 647±5 K doesn't significantly affect the value of n for AMA Al<sub>87</sub>Y<sub>4</sub>Gd<sub>1</sub>Ni<sub>4</sub>Fe<sub>4</sub>. This can be explained by the model of the free volume formed by Y atoms (r = 0.181 nm) with the main component of Al (r = 0.143 nm) that affects mechanical properties. As a result of thermal microhardness increases from 0.20 to 2.75 GPa and during heat treatment for 60 min. at T<sub>3</sub> = 645±5 it decreases to 0.35 and 0.45 GPa for AMA Al<sub>87</sub>Gd<sub>5</sub>Ni<sub>4</sub>Fe<sub>4</sub>. Tensile tests showed that this value for AMA Al<sub>87</sub>Y<sub>4</sub>Gd<sub>1</sub>Ni<sub>4</sub>Fe<sub>4</sub> (fig.1).



Figure 1. The graphs from the tensile test for initial AMA: 1-Al<sub>87</sub>Gd<sub>5</sub>Ni<sub>4</sub>Fe<sub>4</sub>, 2-Al<sub>87</sub>Y<sub>4</sub>Gd<sub>1</sub>Ni<sub>4</sub>Fe<sub>4</sub>

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