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ON MECHANICAL BEHAVIOUR OF PRESSURE-ASSISTED, SINTERED Al-Mg COMPOSITE

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Keywords: Al-Mg composite, mechanical behaviour, porosity, finite element simulation

Abstract: Lightweight materials like Al-Mg composite are attractive especially for aerospace or automotive industry. Current paper investigates mechanical behaviour of hot-pressed, reactive sintered Al-Mg composite with initial Mg volume content of 60% using finite element simulation. Conducted numerical simulations study effect of the porosity on mechanical behaviour. Overall porosity (in percent) is decisive factor for mechanical behaviour of investigated Al-Mg composite rather than number or size of pores.

1 Introduction

Lightweight materials like Aluminium (Al)-Magnesium (Mg) composite are in special attraction of automotive and aerospace industry [1-4]. Al-Mg composite combines excellent density of Mg (1.73 g/cm^3) with Al corrosion resistance (density of Al is 2.7 g/cm^3) [2,4].

Particulate reinforced metal matrix composites (MMCs) have isotropic properties, easier processing route than other metal matrix composites and they are cheaper than other metal matrix composites [3]. MMCs (with various reinforcements) can be produced by casting, metal infiltration, friction stir welding or powder metallurgy [3]. Powder metallurgy has an advantage in controlling matrix and reinforcement properties like shape and size of the reinforcement particles, particle distribution and volume fraction in the matrix [3]. However, an inherent characteristic of powder metallurgical components is porosity that influences mechanical behaviour [5-7]. Generally, porosity is characteristic of many engineering materials like concrete [8-11] or soils [12-16].

One promising technology how to deal with porosity in powder metallurgy is the usage of pressure-assisted reactive sintering [17] - practically porous free samples with relative high density were produced for instance in [18,19].

Pressure-assisted reactive sintering was used to produce many (also non-metal) composites such as $\text{ZrB}_2\text{-SiC-ZrC}$ [20], $\text{B}_4\text{C/Li}_2\text{O-Al}_2\text{O}_3\text{-SiO}_2$ [21], Al-Si [22], $\text{ZrB}_2\text{-SiC-ZrO}_2$ [23], $\text{TiC/Ti}_3\text{SiC}_2$ [24] or reinforced Al matrix composites [3] [25]. Depending on material and processing conditions, porosity varied between 2.4% [21] and 26% [24]. Al-NiO composite had a porosity between 5-8.7% [25] and no porosity measurement was conducted for Al-Mg composite in [3]. Contrary to discussed effect of the porosity on the mechanical behaviour in additive manufacturing (AM) [26-27], it seems to be relatively lack of the information about the quantitative effect of the porosity on the mechanical behaviour of the Al-Mg composite produced by pressure-assisted reactive sintering. Therefore, the aim of the paper is studying effect of the porosity on the mechanical behaviour of Al-Mg

composite. Mechanical behaviour of Al-Mg composite with initial Mg volume content of 60% is investigated using FE simulation.

2 Finite element and material model

2D finite element (FE) model consists of one square shaped part with size of $100 \times 100 \mu\text{m}$ with plane strain thickness of $100 \mu\text{m}$. Pores are embedded in the model as free space in the solid volume. Pores positions are created with Python numpy library with uniform distribution over the size of the FE model ($100 \times 100 \mu\text{m}$) and imported to FE software Ansys. The size of the pores is chosen to produce 1% and 2% porosity in the FE model. 10 and 20 pores are created in the FE model with diameter d approximately $3.57 \mu\text{m}$, respectively $5.05 \mu\text{m}$. FE model is fixed at the bottom edge and loaded on the top edge with tensile loading 400 MPa . Mesh element size is $1 \mu\text{m}$ in the whole FE model. Scheme and size of the FE is shown in Figure 1.

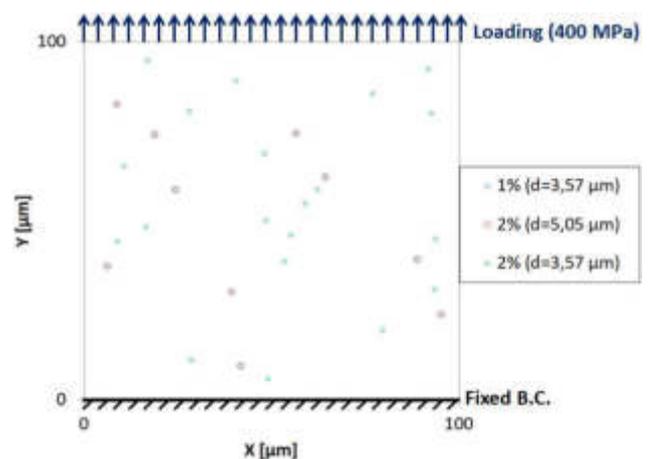


Figure 1 Scheme and size of the FE model. Legend describes the size of the pores in the FE model. 1% and 2% porosity is created in the FE model by varying the pore diameter d and number of pores. FE Model is loaded on the top edge with tensile load 400 MPa

Material model is based on the measurement presented in [3]. There were Al and Mg powders blended by

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planetary ball mill at rotational speed 100 RPM under argon atmosphere for 1 hour, hot-pressed at temperature 673 K under pressure 640 MPa for 10 minutes under Argon atmosphere and reinforced with Al_3Mg_2 and $Al_{12}Mg_{17}$ intermetallics produced during the pressure-assisted sintering process. Details about the production process and produced microstructure are described in the referenced paper [3].

Figure 2 shows material model of Al-Mg hot-pressed, reactive sintered composite with initial Mg volume content $V=60\%$ taken from [3] (Denotes as “Shahid2018” in the legend).

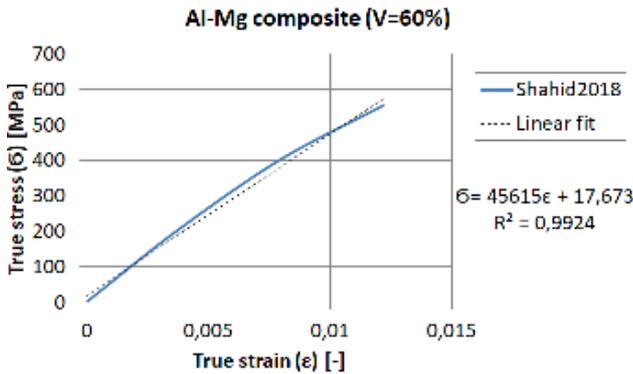


Figure 2 Al-Mg hot-pressed, reactive sintered composite with Mg volume content $V=60\%$. Measured data is taken from [3]. Measured data is linearly fitted with a resulting coefficient of determination R^2 greater than 0.99

Measured data is linearly fitted with a resulting coefficient of determination R^2 greater than 0.99. Value 45615 represents according to Hooke’s law Young’s modulus E . Hence, the material behaviour is (almost) linear elastic, the linear-elastic material model is used in the FE simulation. The yield strength σ_y and tensile strength σ_{TS} are both set on 574 MPa (see Figure 2).

3 Results

Stress behaviour is for comparison expressed by safety factor simply as $F=\sigma_1/\sigma_{TS}$ (Maximum tensile stress failure theory), where σ_1 is maximal principal stress. It can be shown that σ_1 corresponds with σ_Y (stress component in the loading direction, see Figure 1). Maximal principal stress criterion has been identified as suitable for describing failure in presence of defects (pores) in brittle materials [26,28].

Presented Safety factor can be viewed only as a comparison measure among presented model variations.

Figure 3 shows Safety factor F distribution for 1% porosity FE model with pore diameter $d=3.57 \mu m$. Distribution of the safety factor around all pores show similar behaviour – Minimum safety factor F is calculated perpendicular to the loading direction. Pores are stretched in the deformation direction (Y-Axis). It can be assumed that crack would start to initiate and growth in Mode I from

the marked position with minimum safety factor where maximum principal stresses occur.

According to given safety factor definition: $F=0.44572$ gives maximal principal stress σ_1 approximately equal to 1288 MPa. Safety factor outside pores lies approximately between 1.2÷1.4 in almost whole FE model.

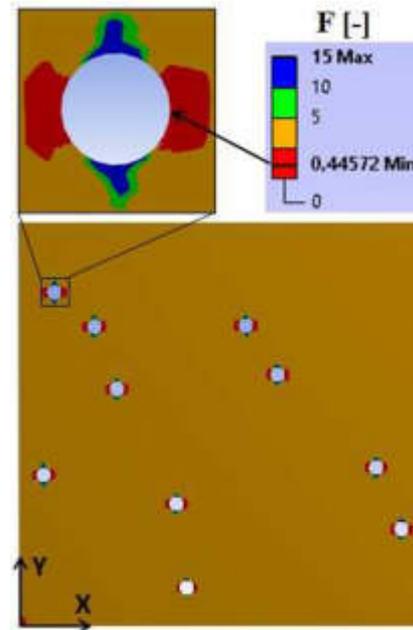


Figure 3 Safety factor F distributions in FE model with detailed view on the most critical position. Minimum calculated safety factor $F=0.44572$ corresponds with maximum principal stresses approximately of 1288 MPa. Safety factor (stress) distribution is similar around all pores in the FE model

Figure 4 shows summarisation of the results. Minimum safety factor F is in free porous FE model (0% porosity) approximately 1.4. It corresponds roughly with maximum principal stresses σ_1 of 400 MPa. 400 MPa is given loading in the FE model - the FE model is verified.

Figure 4 shows a minimal safety factor for 0% (porous free FE model), 1% and 2% porosity. Free porous FE model gives the highest safety factor almost 1.4 and the smallest safety factor 0.365 is calculated for 2% porosity with 20 pores ($d=3.57 \mu m$). Pores reduce safety factor significantly going from free porous FE model to 1% porosity. Porosity is changed negligible between 1% and 2% porosity. Values are taken from whole model and a minimum value is not located in the one position.

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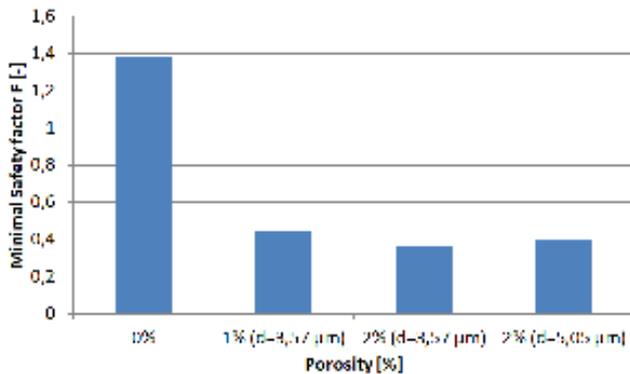


Figure 4 Minimal Safety factor F for 0% (free porous FE model), 1% and 2% porosity. 2% porosity with 20 pores $d=3,57 \mu\text{m}$ gives the smallest safety factor. The highest safety factor is calculated for free porous FE model

Figure 5 shows Maximal displacement U_y in the Y direction (see Figure 3) for free porous FE model, 1% and 2% porosity. Highest displacement is calculated for 2% porosity with 10 pores ($d=5.05 \mu\text{m}$). Smallest displacement is calculated for free porous FE model.

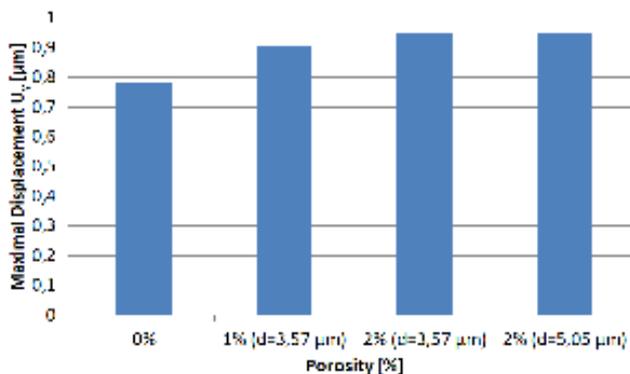


Figure 5 Maximal displacement U_y for 0% (free porous FE model), 1% and 2% porosity. 2% porosity with 10 pores $d=5.05 \mu\text{m}$ gives highest displacement. Smallest displacement is calculated for free porous FE model

Figure 4 and 5 show significant difference between free porous FE model and porous FE models. Stress behaviour (expressed through Safety factor F in Figure 4) demonstrates bigger differences than displacement (Figure 5) among free porous FE model and porous FE models. Differences between 1% and 2% porosity are less pronounced and no conclusion can be made about number and size of pores (2% porosity). Only the overall porosity (in %) regardless size and number of pores is decisive for the mechanical behaviour of Al-Mg composite with initial Mg volume content $V=60\%$.

Conclusions

This paper investigates effect of porosity on the mechanical behaviour of hot-pressed, reactive sintered Al-Mg composite. Al-Mg composite with initial Mg volume content of 60% has been investigated by means of finite

element simulation. Simulations show that overall porosity (in %) has a more pronounced effect on the mechanical behaviour than number or size of pores. The effect is more prominent for stress behaviour (expressed through Safety factor F) than for displacement.

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INVESTIGATION OF MECHANICAL PROPERTIES OF RECYCLED POLYVINYL BUTYRAL AFTER TENSILE TEST

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Keywords: polyvinyl butyral, PVB, safety glass, windshield

Abstract: The paper is focused on experimental testing of mechanical properties of recycled polyvinyl butyral. After homogenization, the mixture polyvinyl butyral was compressed to the test pieces of prescribed shape and size under action of pressure and heat. Polyvinyl butyral foil is one of the most important parts of the windshield or safety glass as interlayer. Recycled polyvinyl butyral was investigated under tensile test. An important consideration is the environmental suitability of materials from recycled polyvinyl butyral and its negative effects on human's life and the environment.

1 Introduction

The implementation of materials from secondary raw materials and their application to possible components reduces the economic and environmental aspects that are also important today [1,2]. The aim of the work is to find areas of use of recycled polyvinyl butyral (PVB) product, which is the product of windscreens recycling. Nowadays, every car and its windshield is equipped with a polyvinyl butyral film that secures the safety of glass. Polyvinyl butyral [3] carries a large amount of chemically bonded energy. Less harmful vapors are released during combustion of PVB than when combustion of heating oil. However, the price of energy obtained from such a waste product is sometimes lower than the price of energy obtained from oil. Due to the way packaging of PVB waste (in the form of "big bags"), it is difficult to handle them. Given the global PVB waste production, the price for this attractive commodity is very reasonable. It ranges from 0,25 € to 0,50 € per kilogram of this thermoplastic. After the PVB separation itself, the waste still contains glass particles that reduce the extent of its use. External storage of PVB material is dissuaded because moisture and ultraviolet radiation generally degrades the properties of PVB [3]. In addition, the possibility of primary PVB contamination is increased. Due to the sticky and soft surface of the recycled PVB film, it is assumed that when unsuitable for storage, the amount of impurities will be easily adhered to the surface, thereby reducing the quality of the use of the recycled product [8]. PVB storage capacities are limited in many countries. Accordingly some countries are already increasing storage costs and are trying to make producers of PVB to encourage, that this type of waste to be reused; they returned it to the production process.

2 Material definition

Polyvinyl butyral (PVB) [4] is a special resin, mainly used as a raw material for laminated safety glass in cars and in the building construction industry (Figure 1). Application is mainly for sky scrapers. PVB currently produces several companies in Europe and the world, each under its trade mark. In addition to the main application and thus the use of PVB films, PVB resins are used for the production of paints, structural adhesives, dry toner paints, and as binders for ceramics and composite fibers [5].



Figure 1 Polyvinyl butyral as a foil in the windshield [4]

The PVB foil has many of excellent features such as high tensile strength, impact resistance, transparency and flexibility, which is particularly useful in producing safety glass. Due to the alcohol, ester and acetate bond content, PVB foil [2] can hold the glass firmly, even if the glass breaks. The glass will adhere to the PVB film interlayer to prevent breakage. Sales of primary end-users of polyvinyl butyral are dependent on the performance of the general economy, especially for safety glass, which is so necessary in the automotive industry and in the construction industry - in architecture. In the market, polyvinyl butyral resins are highly concentrated and are the domain of four companies - Eastman, Sekisui, DuPont and Kuraray [5,6].

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2.1 Recycled polyvinyl butyral

The key material is polyvinyl butyral, which was obtained after recycling windshields of cars (Figure 2). The material has been provided by Schirmbeck GmbH, Germany. The flakes have size from 2 mm to 20 mm and the thickness is from 0.5 mm to 1.5 mm.



Figure 2 Recycled polyvinyl butyral [7]

This recycled polyvinyl butyral is contaminated with dust, glass fragments, so it is important to thoroughly wash the material and dry it before starting laboratory work. Polyvinyl butyral as thermoplastic material is soluble in ethanol, butanol, ethyl acetate, butyl acetate, in a mixture of chlorinated hydrocarbons and insoluble in aliphatic hydrocarbons (in gasoline).

3 Material testing

Before to moulding, the thermoplastic material was homogenized using a double screw machine [8-10] Continuous mixing was used to prepare the homogenized mixture [11,12]. Homogenization (Figure 3) of the material was carried out on a Brabender Plasti-Corder W 350 E [13,14]. Laboratory investigations were performed at room temperature 22 °C and 60% humidity.

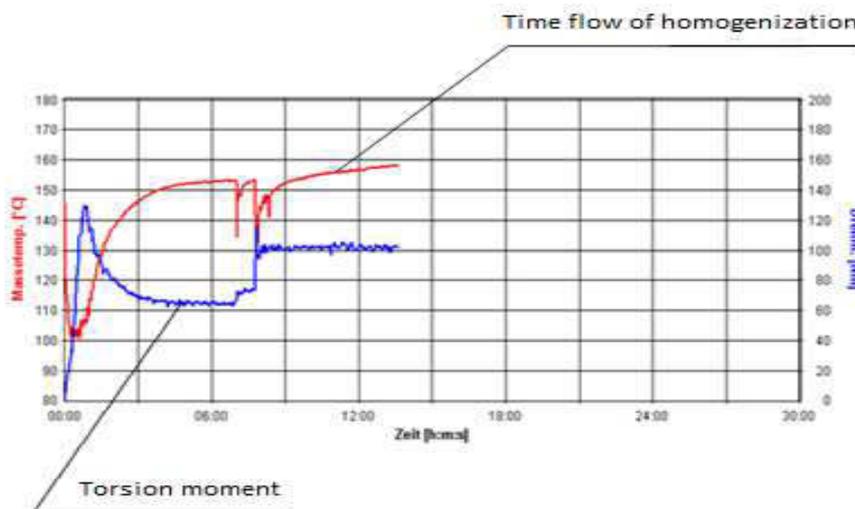


Figure 3 Polyvinyl butyral homogenization [9]

The homogenization process lasted 15 min. The material was thoroughly mixed and the formation of air bubbles that were undesirable during the molding process (occurring during compression, incomplete mixing, or incomplete filling of the thermoplastic mold cavity) was avoided. After homogenization of the recycled polyvinyl butyral on a Brabender Plasti-Corder W 350 E, the material was carefully selected and prepared for molding. The molding cycle [15] was composed of the following operations:

- mold form opening,
- filling the mold with a material,
- closing the mold,
- molding itself,
- mold form opening,
- selection of the mold,

- cooling the mold,
- cleaning the mold [11,12].

The following table (Table 1) shows the molding characteristics used Brabender W 350 mold equipment.

Table 1 Molding characteristics

Equipment	Brabender W 350
Molding temperature	190 °C
Pre-heating time	20 min
Molding time	20 min
Cooling time	20 min
Molding pressure	10 MPa

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As mentioned above, after completion of the homogenization, the test samples were pressed according to DIN EN ISO 527-1 [12,13]. The tensile test (Figure 4) evaluated the tensile strength of the material (Table 1).

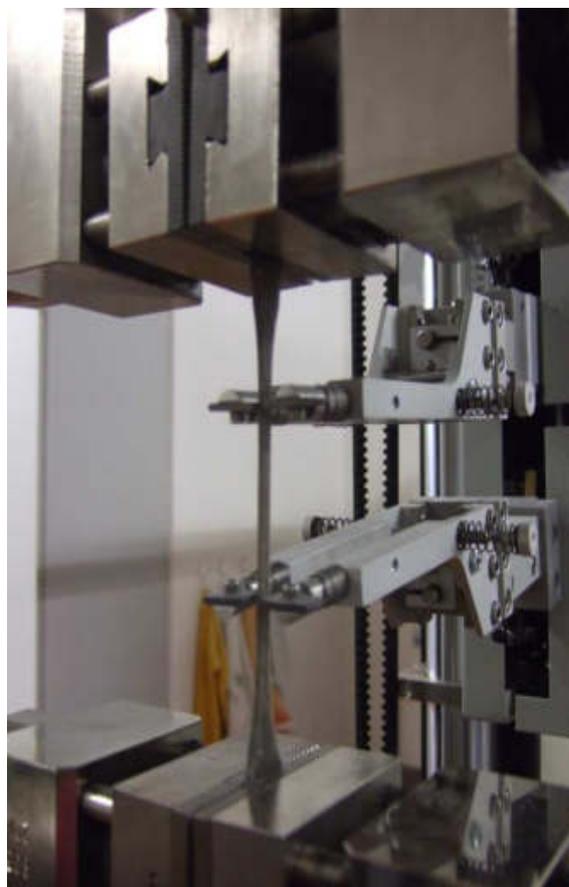


Figure 4 Tensile test of recycled polyvinyl butyral [9]

By tensile test it is important to know an external factors include:

- the frequency and condition of the load of the test material,
- medium stress when material is loaded,
- the stress state of the material,
- aggression and temperature of the environment in which the measurement is taking place,
- the material load history (whether initial testing of the material or repeated load cycles),
- samples geometry,
- test samples material,
- existence or assumption of cracks in materials,
- surface properties of the material,
- method of preparation of the test specimen [11].

Internal factors include:

- chemical composition of the material (FT IR method used),
- structural changes of material,

- method of material processing,
- method of storing the filler in the material matrix.

Table 2 Mechanical characteristics of recycled polyvinyl butyral after a tensile test

	E-modul [MPa]	σ_B [MPa]	ϵ_B [%]	σ_{max} [MPa]	ϵ_{max} [%]
\bar{x}	5,0	17,23	146,14	17,51	145,96
s	2,0	1,47	11,59	1,63	11,64
v	52,4	8,34	7,93	9,34	7,98

The test principle consisted of stressing the test body until the specimen breaks. Due to the constant speed, it has been stretched.

Conclusions

In the current work, the testing of recycled polyvinyl butyral was investigated under tensile test and material stress relaxation. Based on results it can be stated that the material had the max. values for a max. tensile stress of 17,51 MPa, the strain of the material subject to tensile strain was 145,96%. Recycled PVB is an important component in the production of new materials, characterized by very good:

- elasticity,
- adhesion to various surfaces,
- good water resistance.

The advantage of the material is high compatibility with other polymers and also very good possibilities in the manufacture of composite materials.

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Abstract: For the past 40 years we have developed a variety of techniques to create scaffolds. Raw materials, mostly polymers, are processed and shaped into different structures depending on various applications in tissue engineering. One of the main obstacles to the correct creation of fully functional tissue substitution is the complexity of the design as well as the manufacturing process itself. The biomaterial scaffold must be designed to perform the function of the native tissue extracellular matrix and still maintain its bioactivity during interaction with patient's body. In this paper we describe the use of scaffolds in tissue engineering in general.

1 Introduction

Every day, thousands of surgical procedures are performed to replace or repair tissue that has been damaged by illness or injury. An evolving area of tissue engineering focuses on the regeneration of damaged tissue. Detached cells from the body which we associate with porous supporting – scaffolds act as a template for tissue regeneration and lead to the growth of new tissue. Tissue engineering (TE) was first defined in 1988 during the workshop National Science Foundation as an application of the principles and methods of engineering and natural sciences, which lead to an understanding of the structural and functional relationships in physiological as well as pathology altered mammalian tissue and to the development of biological substitutes so as to restore, maintain or improve function [1].

Disease, injury or trauma can lead to damage and degradation of tissue in the human body, requiring treatment for repair, replacement or regeneration. Treatment usually focuses on tissue transplantation from one site to another in one patient (autograft) or from one person to the other (allograft). While such treatment is revolutionary and life-saving, there are fundamental problems with both techniques [1].

While such treatment is revolutionary and life-saving, there are fundamental problems with both techniques. Autographs are expensive, painful, limited by anatomy, and associated with donor disease due to infection or hematoma. Similarly, it is also true for allografts and transplant because of the potential risks of rejection of the transplant by the patient's immune system and the

possibility of introducing an infection or donor disease to the patient. The field of tissue engineering focuses on the regeneration of damaged tissues rather than replacing them, the development of biological replacements for recovery, preservation or improvement of tissue function [1-4].

The field of tissue engineering is a multidisciplinary area that draws on the knowledge of clinical medicine, engineering, material science, genetics and related disciplines, as well as natural sciences and engineering. This area is largely based on the use of 3D porous scaffolds to provide a suitable environment for the regeneration of tissues and organs. In essence, these scaffolds act as a template for tissue formation and are usually deployed by cells and growth factors, or are subjected to biophysical stimulus in the form of a bioreactor, device or system that applies various types of mechanical or chemical stimulation of cells [1, 5, 6]. These cell-based scaffolds are either cultured for *in vitro* tissue synthesis, which are then implanted directly into the damaged site or implanted directly into the site of *in vivo* damage where the regeneration of tissues or organs is stimulated by the body's own system [7]. This combination of cells, scaffolds and signals is often referred to as the "triple or trio of tissue engineering" (Figure 1). The term scaffolds refers to a 3D material before it was embedded with cells (*in vitro* or *in vivo*) [1, 8].

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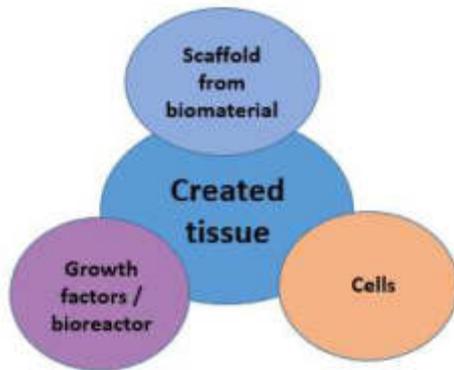


Figure 1 "Trio of tissue engineering" [1, 9]

1.1 Scaffolds and cells

In order to allow cellular morphogenesis associated with living tissue functionality, cell support is necessary to support the cells in their physical 3D support structure. Without adequate 3D support, cell formation can be achieved in only one layer of tissue. Cells without 3D support will not begin to create multiple layers in 3D space in the form of tissue. This phenomenon is known as "contact inhibition", characterized by the limitations in the design of complex tissue structures of the 2D supports such as for example the tissues from the culture flask. Therefore, cells are seeded and cultured in 3D porous structures known as scaffolds to improve tissue regeneration. Scaffold works as a temporary extracellular matrix (ECM), which is intended for the arrangement of cells in 3D architecture and to provide incentives necessary to produce the desired tissue. The resulting substitute of living tissue is composed of two basic parts: living cells and a suitable material that is able to temporarily function as the ECM [10, 11]. The cells may be isolated from the patient's body and then grown on the scaffold *ex vivo* prior to implantation or received *in situ* from the healthy tissue surrounding the site of implantation. The most commonly studied cells in connection with scaffolds are mesenchymal stem cells [12, 13]. Scaffolds should be able to support cells with respect to structural integrity, provide them with sites for adhesion and allow cell morphogenesis and migration, which are key factors in the tissue regeneration process (Figure 2).

One of the main obstacles that prevent proper creation of a fully functional replacement tissue is the complexity of the design and actual production biomaterial scaffold to perform the function of native tissue ECM and still maintain their bioactivity during the interaction with the patient's body. This bioactivity includes the support of the cell remodeling process taking place within scaffolding and the synchronization of remodeling with the ongoing process of tissue repair. Such synchronization should optimally lead to a complete replacement of implanted scaffold that has been created by living tissue, with a consequent degradation of temporary scaffolding. The main focus of tissue engineering is the creation of scaffolds

to provide adequate 3D space for the remodeling of natural tissue, leading to complete replacement of functional living tissue.

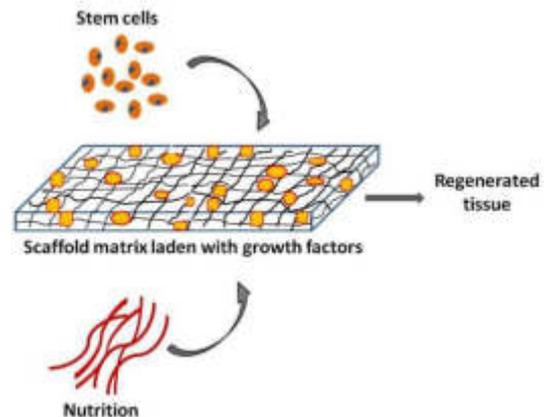


Figure 2: Demonstration of seeding of cells to scaffold [14]

The development of such a complex and sophisticated scaffolding is necessary to apply multidisciplinary scientific platform, which includes the fields of cell biology, developmental biology, biomaterial engineering and advanced manufacturing technology [15, 16].

From the point of view of scaffolding, many studies are focused on supporting tissues such as bones, cartilage, tendons. Bones are among the most intensively studied biological tissues in terms of mechanical properties.

There are three important areas where it is important to know the mechanical properties of tissues:

1. Basic modelling of biological tissues.

Advanced material models are needed to describe the mechanical response of biological tissues to multi-axis load. This mechanical response is partly due to the heterogeneity of mechanical properties, anisotropy, time-dependent mechanical behavior, the presence of several phases (liquid, solid, etc.) and the adaptation of the mechanical properties to the mechanical load. It is important in the development of mathematical models of tissues [17].

2. Regeneration of tissues

Regeneration of damaged tissue by TE and regenerative medicine is an important approach in biomedical engineering. It is necessary to ensure the correct environment for tissue regeneration including the media (i.e. scaffolds, gels, etc.), which are mechanically strong to support the regeneration process. At the same time scaffolds should not be too rigid, because they can otherwise prevent regeneration of the tissue. The question arises, what is the optimal range of mechanical properties of scaffolds and gels? One possibility is to characterize the native tissue and thus get a preview of the expected range of mechanical properties of the scaffold. The properties of

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native tissue are in many cases the best starting point. Moreover, the mechanical properties of biological tissue could be used to diagnose disorders and diseases that are manifested precisely in relation to changes in the mechanical properties of tissue (Figure 3) [18].

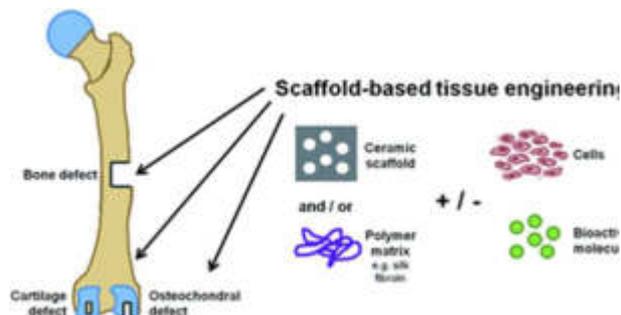


Figure 3: A schematic representation of the use of scaffolds for tissue engineering [19]

3. Tissue damage and trauma

Mechanical stress combined with tissue disease could lead to tissue damage. It is not only the non-physiological stress that occurs in traumatic events but also the physiological load of tissues if chronic damage is present such as for example osteoporosis in bone tissue. We should know what level of load, bones with osteoporosis still tolerate, without the risk of fracture. Similarly, in the study of osteoarthritis it is important to compare changes in mechanical properties of cartilage. We know that changes in the cartilage is one of the first indicators of the onset of osteoarthritis [20, 21].

2 Conclusion

Creating a scaffold with the required properties such as mechanical strength and chemical properties of surface controls tissue regeneration. These properties can be modified and adapted by a suitable choice of material, scaffold's components and, in particular, by the manufacturing technique itself.

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