SSRG International Journal of Mechanical Engineering
ISSN: 2348-8360/ https://doi.org/10.14445/23488360/IJME-V11I1P104

Volume 11 Issue 1, 39-44, January 2024

Original Article

Mechanical, Thermal, and Morphological Analysis of Polyamide-12 and Portland Cement's Composite

Saleh Ahmed Aldahash

Department of Mechanical and Industrial Engineering, College of Engineering, Majmaah University, Al-Majmaah, Kingdom of Saudi Arabia.

Corresponding Author : saldahash@mu.edu.sa

Received: 15 October 2023 Revised: 21 November 2023 Accepted: 12 December 2023 Published: 20 January 2024

Abstract *- The specimens made of polyamide-12 and Portland Cement mixture were prepared using Selective Laser Sintering (SLS) of Lisa PRO Sinterit Studio to test its mechanical testing in response to a laser power parameter. The polymer-cement mixture powder was sintered into a compact structure to form durable, strong, and functional plastic parts by using SLS. SEM and thermogravimetric analysis were used to determine the composite's morphology and thermal properties. With the aid of a VORTI-SIV pilot sifter machine, a homogenous powdered mixture material of polyamide-12 and Portland Cement was created. An SLS system slices the specimen thickness into 0.075 mm layers to produce the test specimen. This is so that test specimens can be manufactured from a 3D-CAD file. This study was conducted to evaluate the optimal laser power that would produce better properties and an improved final product. Results showed that increasing laser power significantly improved the prepared specimen composite's tensile, compressive, and flexural strength.*

Keywords *- Polyamide-12, Portland Cement, Laser power, Mechanical testing, Properties.*

1. Introduction

Polymers undoubtedly influence the development of nanocomposites. A wide variety of topics are being researched in polymers within nanotechnology. These compounds exhibit unique physicochemical properties not available through the action of their components alone. Over the past few years, scientific interest and industrial applications in polymers have increased. As a result of their wide range of applications, polymers are used widely across various industries, including automotive, food and beverage packaging, energy generation, and storage. Polymers prepared from carbon and layered silicates enhance the properties of polymers [1].

Polymers have different nanostructures based on their mass, chemical structure, semi-crystalline nature, chemical and thermal solubility, and surface area. In recent years, polyamides, polyvinyl alcohols, and polycarbonates have been among the most popular and effective polymeric materials.

Polyamide (PA) is one of the most robust and durable engineering plastics. As a semi-crystalline material, PA is resistant to heat and chemicals. Many forms of PA exist, such as powders, blocks, rods, sheets, and granules. There is a natural source of PA as well as a synthetic source. A polyamide polymer can be classified into four categories, namely polyamide 6, polyamide 11, polyamide 12 and polyamide 66. Due to its extraordinary properties, polyamide-12 (PA-12) can be used for a wide range of applications.

The findings of Horgines et al. suggest that polyamide-based waste powder can be lightweight aggregates [2]. Additionally, Guler found that hybridized PA fibers in concrete have greater strength than micro- and macro-PA fibers alone [3]. The results of the previous study demonstrated that combining white cement with PA12-sintered specimens could significantly improve their mechanical properties.

An additional study examined the influence of scanning vector length and EOV effect on the mechanical and morphological properties of PA12/white cement mixture SLS parts. PA-11 and nanocomposite films were studied for their morphology and mechanical behavior under thermal treatment. The addition of Cellulose Nanofibers (CN) increased tensile strength and modulus both before and after treatment, but the changes were more significant in the treated films.

PA-11 films treated with five wt% (CN) showed the best mechanical properties, with 40% higher Young's modulus and 35% higher tensile strength than the matrix [4]. The hydration of cement causes mass concrete to experience thermal stresses. While mass concrete is being placed, temperature differences may occur. It has been found that using PAs at different temperatures and levels of water absorption varies significantly in their mechanical properties. It is highly hygroscopic because PA contains carbonyl and hydroxyl groups in its polymer chains [5]. The influence of mechanical recycling on the properties of recycled polyamides and their microstructures has recently been reviewed [6].

The SLS method is widely used to prepare polyamide 11 and 12 powders at temperatures ranging from 150°C to 185°C [7, 8]. The properties and microstructure of laser-sintered plastic products depend on several process and material parameters. Various thermoplastic materials have been developed using SLS, including PA-12. However, It is possible to enhance the mechanical properties of fabricated specimens by accumulating rigid particles in polymers, including greater flexural strength, compression strength, rigidity, toughness, resistance, and, occasionally, tensile strength.

This work aims to increase the mechanical properties of sintered specimens by adding cement to PA-12 and reducing the product cost since PA-12 is relatively expensive. Variations in laser energy density are investigated to determine the prepared composite's morphology, thermal properties, and mechanical properties. To obtain high-quality parts and new SLS materials, it is essential to understand how material properties relate to SLS parameters. The laser power can be changed at fixed scan spacing and laser speeds to control the laser energy density.

The mechanical properties of sintered specimens were investigated by analyzing fabricated specimens' tensile, flexural, and compressive strengths. Following these findings, the present work examines the different stages of PA-12 and Portland cement composite sintering. This paper discusses the thermal and morphological properties of PA-12 and Portland cement mixture. The effect of laser power on the prepared specimen was tested to check its various mechanical properties. By understanding how SLS parameter settings relate to material properties, manufacturing parts with predetermined properties will be possible.

In the previous literature, polyamide-6 was mainly used to prepare composites and test their mechanical properties. It has been demonstrated that polyamide-12 and cement composites can be prepared and subjected to various automated tests. Still, white cement was used in those studies, whereas gray cement was chosen in this study. Furthermore, this study used a different SLS machine than the previous ones. Also, the laser power range was diverse. This helps the author achieve better and new results compared to existing literature.

2. Materials and Methods

Cement-filled PA12 parts are manufactured using the SLS technique in the following sections. In addition, the testing methods and operating conditions are described.

2.1. Materials

In this study, polyamide-12 was added to a Portland Cement sample and used as a powdered mixture. Portland Cement (PC) was acquired from a local Al-Majmaah City, Saudi Arabia store. Portland cement is high-quality cement made from high-quality clinkers. In construction, PC is a variety of high-quality materials. Its production quality was tested per Saudi quality standards (SASO GSO 1917/2009). Portland Cement is used to bind solid masses together. Typically, it is composed of limestone, chalk, and clay, mainly calcium oxide, silica oxide, aluminum oxide, and iron oxide.

To solve the problems of high-temperature compatibility and chemical resistance, a polyamide-12 powder with excellent mechanical properties and chemical resistance was purchased from commercially available Sinterit (Poland). The obtained polyamide-12 powder is gray and has a melting point of 185°C, as specified by the company.

A powder composed of polyamide-12 consists of rounded particles, a narrow particle size distribution, and ultrafine and non-crystalline properties. In addition to being safe to use with food, polyamide-12 is also harmless to the environment. Table 1 lists the specifications of used PA-12 and Portland cement powder. It can be seen that PA-12 and Portland cement powder specifications differ.

Table 1. Specifications of PA-12 and Portland Cement powder

Powder Properties	PC	PA-12
Mean Grain Area (μm)	15	50
Apparent Density (g/cm³)	1.2	0.5
Particles Dimension	Non-Uniform	Non-Uniform
Melting Point (°C)	1400	185

2.2. Fabrication of Powdered Mixture

Sinterit's ultrafine PA12 smooth powder is mixed with Portland cement at a specific weight fraction (90% of PA-12 and 10 % cement) to produce a specimen containing a particular fraction of cement by weight. A VORTI-SIV pilot sifter was used to vigorously shake the Portland Cement and polyamide-12 mixture to prevent agglomeration.

Powder mixtures were instinctively mixed in a high-speed electrical blender for 25-30 minutes according to a predetermined formulation to obtain homogeneous powder mixtures and uniform colors.

2.3. SLS Process of PA-12/ Cement Composite and Characterization

The powder mixture is sifted and homogenized, then put into the container of the “Sinterit Studio 2019” machine, according to Table 2. An SLS system slices the specimen thickness into 0.075 mm layers to produce the test specimen. This is so that test specimens can be manufactured from a 3D-CAD file by scanning the powder bed with the laser head. When the scan is completed, an additional layer of powder mixture is laid on top of the sintered layer. The process is repeated until the entire thickness of the sintered specimen has been achieved.

Laser Power levels (LP) of 1 to 3 watts were used to manufacture the test specimens. A soft brush and sand plaster machine clean the sintered part from the part build, then mechanically remove any affixed powders. Agar auto sputter coater is then used to coat the specimens with an ultrathin layer of gold to be inspected using a Scanning Electron Microscope (SNE-4500M Plus, South Korea). An analysis of the thermal strength and effect of temperature on the powdered mixture was conducted using thermogravimetric analysis using Simultaneous Thermal Analyzer, Perkin Elmer, USA.

Table 2. Specifications of Sinterit Studio 2019 machine

Model	**Lisa PRO Sinterit Studio 2019**
Process	Selective Laser Sintering
Laser Category	IR Laser Diode
Laser Power (Watt)	3
Build Speed (mm/h)	3
Print Volume (mm)	110 × 160 x 230
Computer System	Windows 11
Power Supply	220-240 Volts, 50-60 Hz

2.4. Mechanical Testing

Testing is then performed on the specimens to determine their tensile, compressive, and flexural strengths. British Standard (references 527.2, 178, and 604) were used as guidelines for preparing the specimens for tensile, flexural, and compressive testing, respectively, following American Society for Testing and Materials (ASTM) standards D0638, D0790, and D0695. Figure 1 shows the geometry and dimensions of the specimens used for tensile, compressive, and flexural tests.

3. Results and Discussion

According to Figure 2, the tensile strength increases rapidly when laser power is increased. The laser level may enhance the binding of PA particles, thereby decreasing porosity and producing a denser structure with a greater tensile strength. Because there is insufficient energy, a small laser power creates a softer, less dense, and tensile part. The more energy the laser generates, the more compact the formed part becomes.

Hou et al. also found that when the laser power increases between 40W and 50W, there is a slight increase in tensile strength [9]. Xu et al. used a fabricated specimen and noted that the tensile strength of a material continuously improves with increased laser energy density, indicating enhanced sintering between adjacent layers and a strengthened curing zone between the adjacent layers [10].

For flexural strength, the results indicate that when laser power is increased, the powder is heated more intensely (Figure 3). In addition to the powder particles targeted by the laser beam, neighboring powder particles also melt. It is shown in the figure that when the laser power increased, it resulted in a reduction in the number of pores of polyamide powder.

As a result, an improved flexural strength was generated due to the compact structure. Negi and Sharma found that by increasing the laser power, the polyamide particles began to neck, and as melted polyamide filled the gaps between the glass particles, the gaps became smaller [11].

In their study, Zhu et al. developed a selective laser sintering method for the preparation of high-performance carbon fibers/polyamide12/epoxy ternary composites, and they demonstrated an increase in flexural strength of nearly 50 MPa for finished parts after selective laser sintering [12]. Increasing laser energy density increased the sintered parts’ average flexural strength to 0.0277 J/mm^2 with a maximum of 2.12 MPa [13].

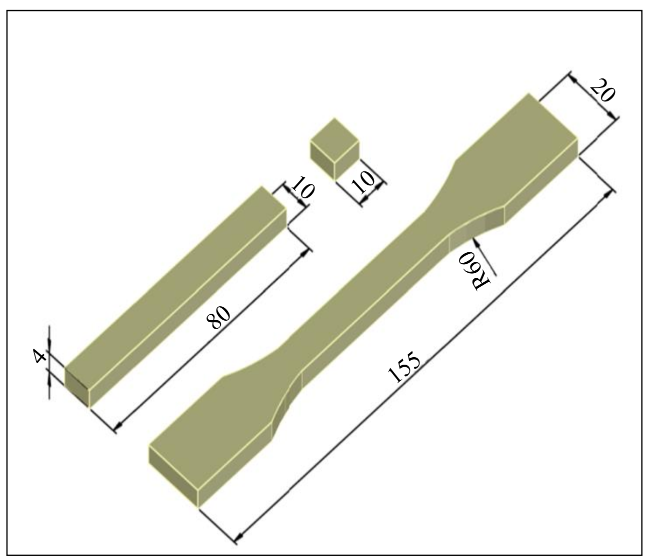

Fig. 1 Geometrical shape of test specimen composites

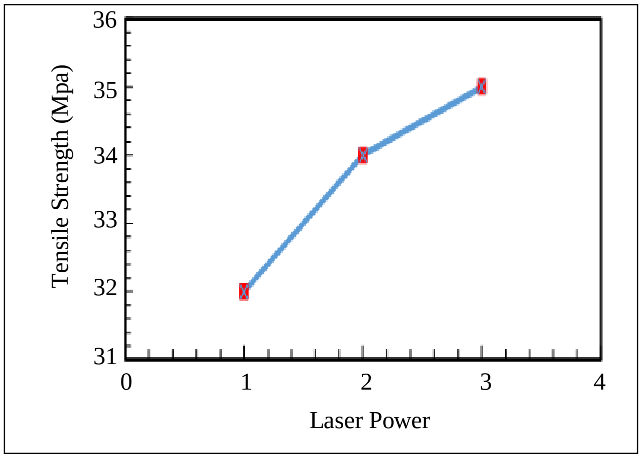

Fig. 2 Effect of laser power on the tensile strength of the specimen

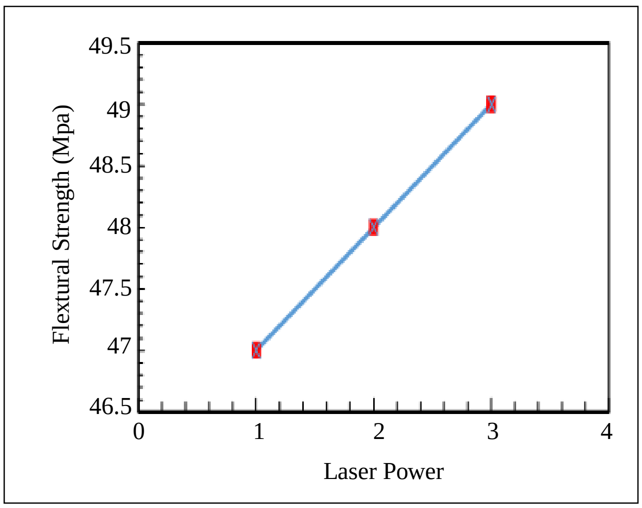

Fig. 3 Effect of laser power on the flexural strength of the specimen

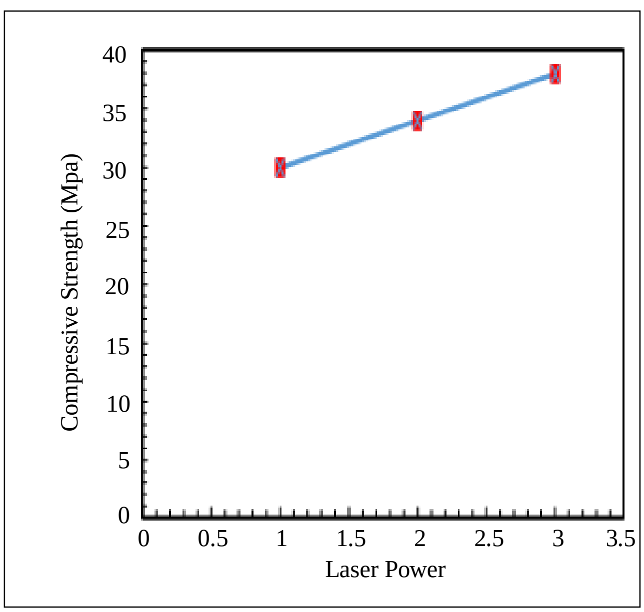

Fig. 4 Effect of laser power on the compressive strength of the specimen

Figure 4 shows the deviation of compressive strength versus laser power for fiber lasers with different powers. When laser power is increased, PA-12/cement composite compressive strength rises considerably. As the high-power laser beam generates more thermal energy, the PA-12/cement nanopowder has shown excellent stability, possibly because the studied laser power range did not affect its compressive strength. The results suggested no degradation of the sintered part and the binder material with increased laser power. Using five lattice structures for compressive testing in a study by Bi et al., the compressive strength increased first, then decreased with laser power, with the determined value occurring at 200W-500 mm/s [14].

3.1. Characterization

The SEM image of the composite shows a smooth surface and homogeneous mixing and distribution of PA-12 and cement particles as a pack (Figure 5). The surface of the composite is porous, contains macropores and micropores, and has regular shapes and gaps. A dense matrix is visible at various magnifications, and the surface is almost unchanged. Softness and holes are pretty evident. Both polyamide-12 and cement agglomerated with each other quite well.

TGA was used to determine the thermal properties of the mixture powder sample. According to the Figure 6, the main change occurred near 500°C due to non-volatile residue degradation [15], and it is obvious that volatiles are lost at high temperatures, causing weight loss. Based on the weight loss curves, it was possible to observe a clear picture of the thermal stability of nanopowder as a function of radiation doses; the cement also slightly impacts the PA12 matrix. The derivative peak temperature (%) is around 489°C, which, on a weight loss curve, the inflection point represents the point at which the rate of change is most excellent.

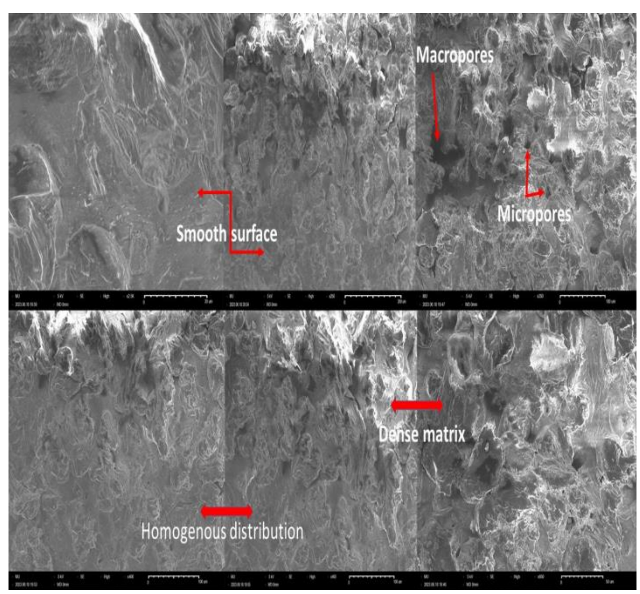

Fig. 5 SEM images of composite polyamide-12 and Portland Cement

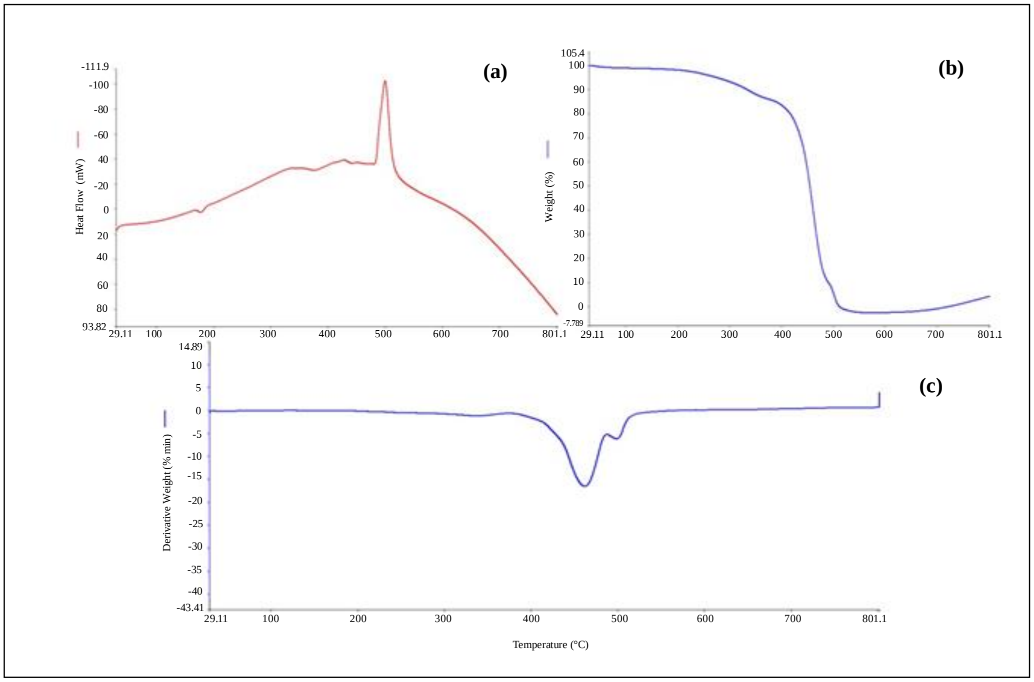

Fig. 6 TGA of polyamide-12 and Portland Cement powdered mixture

4. Conclusion

This study evaluated the effect of laser power on the tensile, flexural, and compressive strength of SLS-made specimens of polyamide-12 and Portland Cement. To produce high-quality parts, it was essential to identify a laser power that would provide better mechanical properties. Different characterization methods were used to study morphological and thermal properties using SEM and TGA. The SEM image of the nanocomposite shows a smooth surface and homogeneous mixing and distribution of PA-12 and cement particles as a pack. The nanocomposite's surface is porous, containing macropores and micropores, and has regular shapes and gaps. As the high-power laser beam generates more thermal energy, the PA-12/cement-made specimen shows excellent stability and improved tensile, flexural, and compressive strength. The results suggested no degradation of the sintered part and the binder material with increased laser power.

References

[1] Michael Alexandre, and Philippe Dubois, "Polymer-Layered Silicate Nanocomposites: Preparation, Properties, and Uses of a New Class of Materials," *Materials Science and Engineering: R: Reports*, vol. 28, no. 1-2, pp. 1-63, 2000. [CrossRef] [Google Scholar] [Publisher Link]

[2] M. Horgnies et al., "Effects of the Use of Polyamide Powder Wastes on the Microstructure and Macroscopic Properties of Masonry Mortars," *Cement and Concrete Composites*, vol. 52, pp. 64-72, 2014. [CrossRef] [Google Scholar] [Publisher Link]

[3] Soner Guler, "The Effect of Polyamide Fibers on the Strength and Toughness Properties of Structural Lightweight Aggregate Concrete," *Construction and Building Materials*, vol. 173, pp. 394-402, 2018. [CrossRef] [Google Scholar] [Publisher Link]

[4] Denis Mihaela Panaitescu et al., "Influence of Thermal Treatment on Mechanical and Morphological Characteristics of Polyamide 11/Cellulose Nanofiber Nanocomposites," *Journal of Nanomaterials*, vol. 2015, pp. 1-11, 2015. [CrossRef] [Google Scholar] [Publisher Link]

[5] Varun Venoor et al., "Understanding the Effect of Water in Polyamides: A Review," *Polymer Reviews*, vol. 61, no. 3, pp. 598-645, 2021. [CrossRef] [Google Scholar] [Publisher Link]

[6] Ichrak Ben Amor et al., "Mechanical Recycling and Its Effects on the Physical and Mechanical Properties of Polyamides," *Polymers*, vol. 15, no. 23, pp. 1-15, 2023. [CrossRef] [Google Scholar] [Publisher Link]

[7] David K. Leigh, "A Comparison of Polyamide 11 Mechanical Properties between Laser Sintering and Traditional Molding," *2012 International Solid Freeform Fabrication Symposium*, pp. 574-605, 2012. [CrossRef] [Google Scholar] [Publisher Link]

[8] R.D. Goodridge, C.J. Tuck, and R.J.M. Hague, "Laser Sintering of Polyamides and other Polymers," *Progress in Materials Science*, vol. 57, no. 2, pp. 229-267, 2012. [CrossRef] [Google Scholar] [Publisher Link]

[9] Gaoyan Hou, Hong Zhu, and Dan Xie, "The Influence of SLS Process Parameters on the Tensile Strength of PA2200 Powder," *IOP Conference Series: Earth and Environmental Science*, vol. 571, pp. 1-7, 2020. [CrossRef] [Google Scholar] [Publisher Link]

[10] Xu Zhifeng et al., "Effects of Laser Energy Density on Forming Accuracy and Tensile Strength of Selective Laser Sintering Resin Coated Sands," *China Foundry*, vol. 11, no. 3, pp. 151-156, 2014. [Google Scholar]

[11] Sushant Negi, Rajesh Kumar Sharma, and Suresh Dhiman, "Experimental Investigation of SLS Process for Flexural Strength Improvement of PA-3200GF Parts," *Materials and Manufacturing Processes*, vol. 30, no. 5, pp. 644-653, 2015. [CrossRef] [Google Scholar] [Publisher Link]

[12] Wei Zhu et al., "A Novel Method Based on Selective Laser Sintering for Preparing High-Performance Carbon Fibres/Polyamide12/Epoxy Ternary Composites," *Scientific Reports*, vol. 6, pp. 1-10, 2016. [CrossRef] [Google Scholar] [Publisher Link]

[13] Yas Khalil, Adam Kowalski, and Neil Hopkinson, "Influence of Energy Density on Flexural Properties of Laser-Sintered UHMWPE," *Additive Manufacturing*, vol. 10, pp. 67-75, 2016. [CrossRef] [Google Scholar] [Publisher Link]

[14] Jiang Bi et al., "Formability, Surface Quality and Compressive Fracture Behavior of AlMgScZr Alloy Lattice Structure Fabricated by Selective Laser Melting," *Journal of Materials Research and Technology*, vol. 19, pp. 391-403, 2022. [CrossRef] [Google Scholar] [Publisher Link]

[15] Sofian Ibrahim et al., "Mechanical Properties and Thermogravimetric Analysis of Peroxide Prevulcanized Natural Rubber Latex Induced by Co-60 γ Radiation," *IOP Conference Series: Materials Science and Engineering*, vol. 1231, pp. 1-8, 2022. [CrossRef] [Google Scholar] [Publisher Link]