*Paper presented at the East Europe Conference on AM Materials (EECAM21)*
*September 2-4, 2021, Belgrade, Serbia*

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# PRELIMINARY STEPS FOR EXPERIMENT PLANNING IN ADDITIVE TECHNOLOGIES STUDIES
# PRELIMINARNI KORACI PLANIRANJA EKSPERIMENATA U ADITIVNIM TEHNOLOGIJAMA

Originalni naučni rad / Original scientific paper
UDK /UDC:

Rad primljen / Paper received: 15.12.2021

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**Keywords**
- 3D printing
- fused deposition modelling (FDM)
- printing parameters
- material indicators
- experimental design

**Ključne reči**
- 3D štampanje
- modeliranje taložnim stapanjem (FDM)
- parametri štampanja
- materijalni pokazatelji
- dizajn eksperimenta

*Abstract*

*Due to urgent requests of optimizing additive manufacturing (AM) printed parts parameters to meet the requirements for applications beyond prototyping or low-key applications (namely, the machine parts under loading), it is necessary to optimize the technology. The quality requirements include (but are not restricted to) strength, rigidity, fatigue resistance, and surface smoothness. The experimental design technique is worth applying to meet these requirements. Some preliminary experimental data and results are shown, as well as reference materials which are useful to the aims of investigation. It is worth mentioning that AM methods themselves are environmentally friendly (saving the amount of required material) as well as the possibility of obtaining desired results with fewer samples by applying DOE).*

*Izvod*

*Usled hitnih zahteva za optimizacijom parametara štampanih delova aditivne proizvodnje (AM) kako bi se ispunili zahtevi za aplikacije koje se nalaze izvan prototipova ili malih aplikacija (naime, mašinskih delova pod opterećenjem), potrebno je uraditi optimizaciju tehnologije. Zahtevi za kvalitetom uključuju (ali nisu ograničeni na) čvrstoću, krutost, otpornost na zamor i kvalitet hrapavosti površine. Primena tehnike dizajna eksperimenta je opravdana, kako bi se ispunili ovi zahtevi. Prikazani su neki preliminarni eksperimentalni parametri i rezultati, kao i referentni materijali koji su korisni u cilju istraživanja. Vredi napomenuti da su same AM metode ekološki prihvatljive (štedi se količina potrebnog materijala) kao i mogućnost dobijanja željenih rezultata sa manjim brojem uzoraka primenom DOE).*

## INTRODUCTION

In the professional language of aircraft manufacturers there is a phraseological unit 'buy-to-fly ratio', which can be understood as 'the ratio of what you bought to what flew', i.e. how much material is purchased and how much actually 'flew' as a part of the aircraft. According to various sources, this ratio is 15:1 or even 20:1 for complex parts. The use of AM and 3D printing allows you to reduce this figure to 1.5-2.0 : 1. In Fig. 1 we show the schematic representation of the 'spiral of history' - that is how 3D ideas have been developing.

Nowadays, the researchers sometimes face the problem of low mechanical and physical properties of products obtained using this technology. One of the modern methods of additive technologies is fused deposition modelling (FDM), performed using a 3D printer. The main working element of such a printer is an extruder that feeds the material to be welded to the heater and nozzle; usually, they present one unit. The following main elements of a 3D printer are a heated worktable and a mechanism for moving the extruder on the worktable. Manufacturing of a product using the FDM technology is carried out inside an insulated chamber, in which the air temperature is maintained within 100 °C. This technology makes it possible to obtain products without significant residual stresses and with the high bond strength of layers.

## EXPERIMENTAL DESIGN

Due to the high requirements for AM products and a large number of influential parameters, the task of optimisation of the technologies is non-trivial. The terms for developing new technology products are short. In this situation, the Design of Experiments (DOE) might be helpful. DOE was first introduced by Sir Robert Fisher in 1926 during the investigation of crop productivity of seeds, which were planted in slightly varied soil conditions /1, 2/. Main concerns in experimental design include the establishment of validity, reliability, and replicability. It aims to find the optimal parameters composition. The main problem here is considering the variability in experimental outcomes. The famous SN (signal to noise proportion) relation was developed with the aims: 1) to find maximum; 2) to organise the experiment in an optimum way. Although many decades have passed since the DOE concept appeared, its spread is prevented for some reasons. On the other hand, the good news is that while studying AM products and AM technologies, many scientists employ this method. Before the creation the plan, we decided to consider varied links among influential factors.

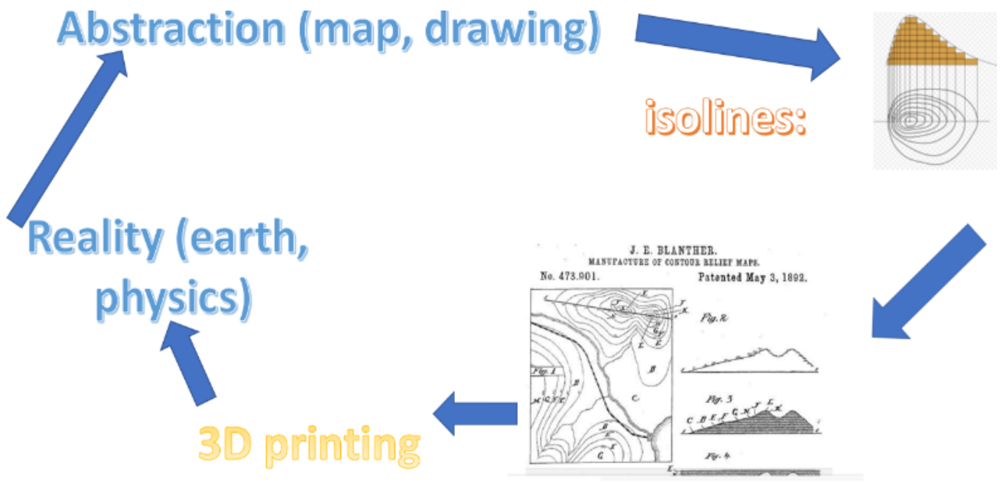


Figure 1. Spiral of history of the scientific thought about 3D during centuries.

## DATA FROM LITERATURE

The paper /3/ contains detailed experimental analysis on thermoplastic lactic acid polymer (PLA) and the impacts of the printing parameters on its strength.

For the experimental study, the samples are printed with two different filling percentages (50 and 100 %). For each filling value, following printing parameters are modified: 4 extrusion temperatures (200, 215, 230, and 245 °C), 4 printing base temperatures (50, 60, 75, and 90 °C) and 3 extruder rates (30, 45, and 60 mm/s). The filling percentage of the sample had the most significant influence on mechanical properties, /3/. Although the data in /4/, as based partly on ANOVA analysis, stresses out the high nonlinearity of influencing factors, the paper /3/ presents linear dependences with high degree of correlation (see also Table 1). Paper /5/ stresses that low-cost FDM 3D printers do not work well automatically but require a calibration phase because the best configuration settings in the slicing software are unknown, and the number of parameter values needs to be manually defined is significant.

In /6/, the problem of the quality of AM products is formulated. The authors discuss the chemical post-processing methods applied to ABS parts produced by FDM that are known to add to surface roughness, dimensional inaccuracy, and staircase effect. These problems are all surface-related issues. They eventually prevent details from being the final product. It is evident that post-processing operations are needed to have ready-to-use parts. The authors discuss the post-processing techniques and categorize them as chemical, mechanical, and thermal post-processing operations. Often the requirements are controversial. In /6/ the importance of low surface roughness is stressed out (especially in the dental industry). However, the searches show that improving the surface quality sometimes negatively correlates with tensile strength characteristics. It is hard to say if this correlation means a causal relationship.

Due to many influential factors, it is worth exploring the particular correlation of the random values (although they are not random by nature, but rather appointed by researchers). The authors of /6/ have observed that some chemical post-processing methods reduce tensile strength and cause deterioration of parts. Considering this situation, it becomes evident that scientists should investigate the quality of parts in a complex way.

Paper /7/ analyses the ultimate tensile strength, dimensional accuracy and surface roughness (SR) of AM. Through analysis of variance (ANOVA) significant factors of each quality characteristic and optimal factors level combinations for each best quality characteristic are obtained. The authors in /7/ employed Taguchi and Gray analysis.

In /8/ to improve the surface quality, three factors of FDM are varied: Layer Thickness (mm) – ‘A’, Part Build Orientation (degree) – ‘B’ and Raster Angle (degree) – ‘C’. A regression equation describing the surface roughness with a high degree of correlation is obtained as follows:

$$Ra = -12.613 + 115.248A + 0.343B - 209.156(A*A) + 0.002(B*C). \quad (1)$$

The study /8/ reveals that the printed parts' orientation has the most significant effect on surface roughness, followed by significance by layer thickness.

In /9/, multi-objective experimentation is implemented where the critical properties of FDM parts include: surface roughness (Ra μm), tensile strength (σ MPa) and ultimate deformation by fracture (Δ %). The researchers chose five parameters /7 / as influential for optimisation: layer thickness (A), air gap (B), raster width (C), contour width (D), and raster orientation (E).

## PRELIMINARY STATISTICAL ANALYSIS

As the first step towards experimental design, we did some statistical investigations. Based on data /9/, the box plot graph (Fig. 2) and pair correlation plot (Fig. 3) are built. Such data representation might demonstrate some discreet relationships: for our particular investigation, the study of strength indicator and its correlation with other factors (Fig. 3) is of great importance.

The results of /9/ encourage us in the possibility of formulating one of the experimental design requirements, namely, the linearity of dependences. For example, the function under regulation (tensile strength $\sigma_b$ [MPa]) depends almost linearly on one of the printing parameters, namely, on the bed temperature $T_b$. With employing the data from paper /9/ it was possible to estimate the coefficients of the linear equation:

$$\sigma_b = A + BT_b \, . \qquad (2)$$

The values of coefficients for two options of filling the matrix are shown in Table 1. The scatter in the dependencies Eq.(2) for both filing percentage options is relatively low.

Table 1. Coefficients in Eq.(2) based on /3/.

| Filling percentage (%) | A | B |
|---|---|---|
| 50 | 23.5 | 0.05 |
| 100 | 44.25 | 0.075 |

It is essential for the aims of future studies to survey the employed optimisation parameters in 3D printing (Y) and the influential factors (X) (active and passive), which are chosen for the control of the experiment. Table 2 shows the 'Y's and 'X's of DOE from various scientific papers.

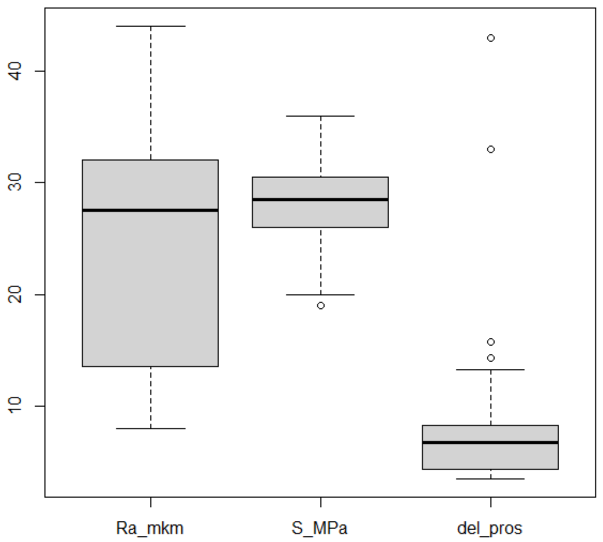


Figure 2. Box-plot of some parameters, obtained in /9/ (Ra µm, $\sigma$ MPa, and ultimate deformation, Δ %).

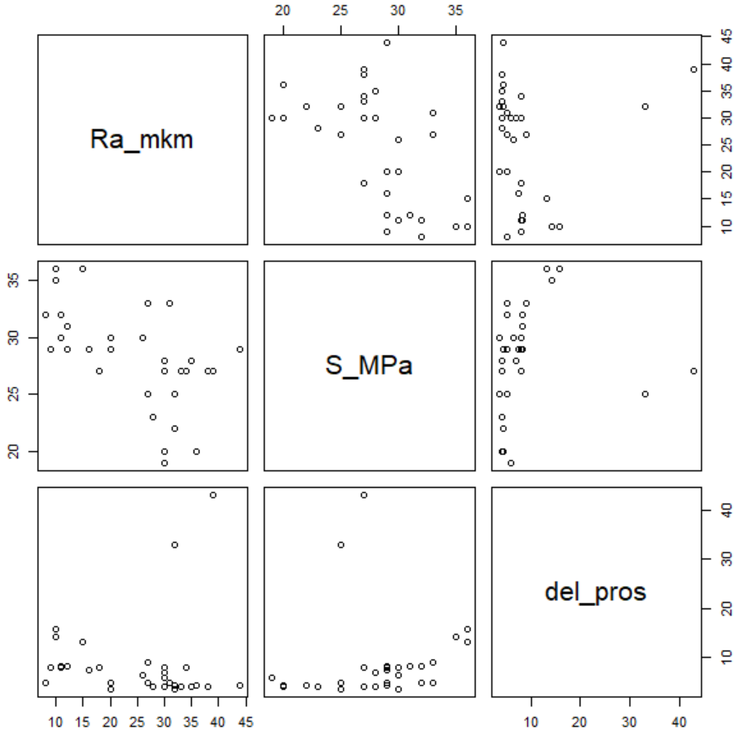


Figure 3. Pair correlation of the experimental results from /9/ (surface roughness (Ra µm), tensile strength ($\sigma$ MPa) and ultimate deformation (Δ %)).

Table 2. Survey based on selected periodicals with DOE in AM.

| Ref. | Paper, year | Y (response) | X (factors) |
|---|---|---|---|
| /3/ | Corrado, 2021 | uniaxial tensile strength | filling percentage; 4 different extrusion temperatures; 4 different printing base temperatures; 3 different extruder rates |
| /4/ | Adanski, 2012 | improving surface | different levels of concentration, time of exposure, temperatures; initial roughness |
| /5/ | Cappetti, 2018 | precision of printing, geometry | extrusion width; infill extrusion width; solid infill extrusion width; top solid infill extrusion width |
| /7/ | Che, 2007 | ultimate tensile strength, dimension accuracy and surface roughness | building strategy, layer thickness and orientation of the part |
| /8/ | Raol, 2014 | response surface methodology (RSM) | layer thickness, part build orientation; raster angle |
| /9/ | Alhubail, 2013 | SR (surface roughness);<br>TS (tensile strength) | layer thickness; air gap; raster width; contour width; raster orientation |

## METHOD

The success of studies like /6, 8/, and some others mentioned before, as well as the urgent necessity to improve the strength indicators of FDM produced parts, has encouraged us to plan the study aimed at strength indicators optimisation. In Table 2 one can see that many of the authors are mainly concerned with the strength, namely, /7, 9, and 3/. Since strength optimisation is the aim, we want to formulate the DOE plan, as recommended in /1, 2/:

– First, we draw a list of the parameters, which are supposed to influent specimens' strength parameter. The list in Table 3 includes controlled parameters (which might be regulated during testing) and parameters, which are just registered. Some of them are already installed by 3D printer manufacturers.
– Second, for each factor in Table 3, the starting point for the experiment and variation limits should be assigned. That allows presenting the values of factors as constants +1 or –1. This step makes it easier to draw the decisive equation similar to Eq.(1).
– Third, initial series of experiments should be performed. Although that is not so evident, in DOE the levels of factors should vary simultaneously and not one by one. Following this procedure, the researcher might sooner reach an optimal combination of factors' levels.

Table 3. Influential factors in 3D printing.

| | |
|---|---|
| Installed factors: | |
| 1 | Print temperature |
| 2 | Layer Speed 30 mm/s |
| 3 | Nozzle diameter 0.5 mm |
| 4 | Filament size 1.75 mm |
| Analysed factor (non-regulated): | |
| Type of plastic: | Grey/White |
| Factors which are varied in the experiment: | |
| Loading rate | 0.015; 0.03; 0.06; 0.04; 0.15 mm/s (depending on the base) |
| Base length | 180, 100, 70, 40, 10 mm |

## EXPERIMENT

Earlier, authors of this paper had investigated strength parameters on parts produced by 3D printing with ABS plastic /10/. The experiment /10/ has shown the advantages of the specimens produced of grey plastics compared with those of white plastic. Figure 4 presents the side view of specimens made of two types of plastic. 'Grey' plastic has a better structural regularity. It also demonstrates better strength parameters, so precisely for this option, further steps are formulated. Also, the experiment reveals that some parameters depend on the experimental procedure. Specimen's length appears to be influential upon limiting contraction and elongation.

The measured hardness depends on where the test is performed: the hardness on the base surface is only 40 % of hardness on the top. These facts should be considered in the research. Examination of the flat surface of white plastic samples under a microscope shows that the surface pattern has lines directed both along the sample and at 45º to its axis. The line thickness is from 290 to 310 μm. Both line thickness and direction are preserved even after sample testing. Figure 4a shows the structure of the sample, consisting of rows of rods (threads) of circular cross-section of diameters 240 to 300 μm, fused along the generatrix. In Figs. 4a, and 4b, one can see the voids formed during the fusion of rods. On the end surface along the sample, layers of thickness 153 to 200 μm are visible.

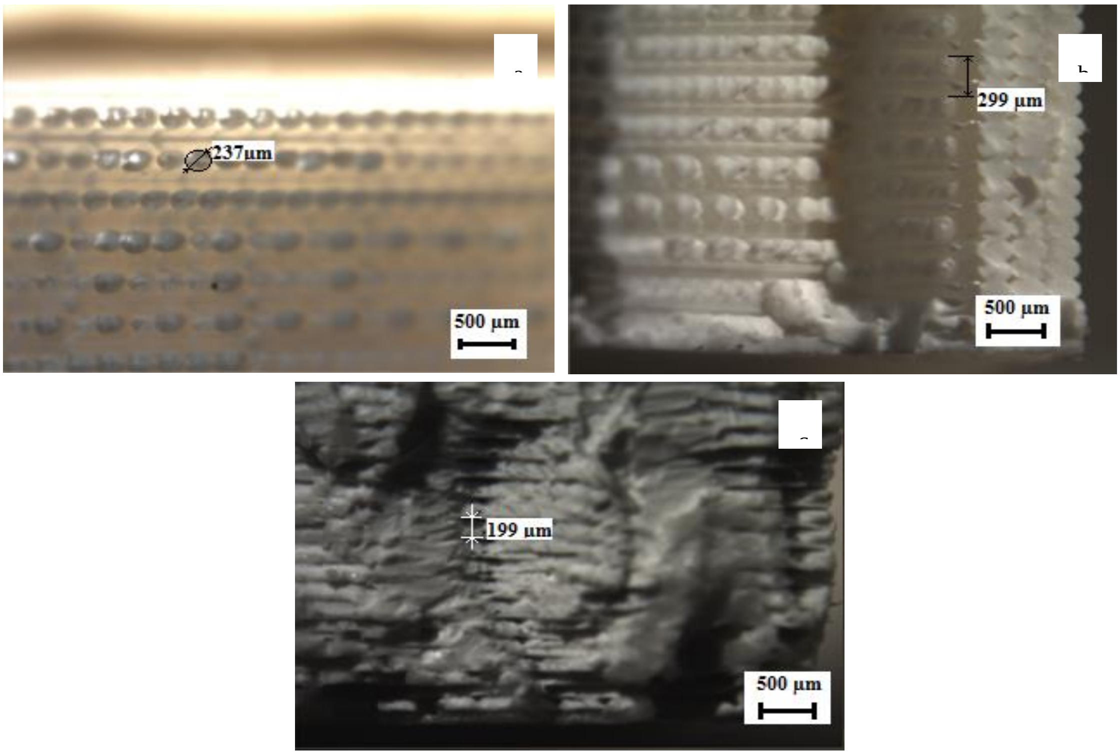


Figure 4. Fracture zone, side view: a) and b) for ABS1P specimen, white plastic; c) ABS2 specimen, grey plastic.

Microscopic examination of the flat surface of a grey plastic specimen shows surface pattern lines both along the specimen and at 45º to the specimen axis. Line thickness is from 0.9 to 1 mm. In this case, both the line thickness and direction remain unchanged after sample testing. Figure 4c shows the location of sample destruction, where the layered structure of the material is visible, while individual fibres are not visible. The layer thickness is 200 μm.

## CONCLUSIONS

Scientists have put a lot of effort into studying AM processes and products. A lot of papers have employed DOE.

An experiment in IMASH RAN reveals some testing peculiarities concerning the topics under investigation.

Advantages of ‘grey’ ABS plastics against ‘white’ ABS plastics is shown experimentally.

The existing 3D printing has several significant technological disadvantages that are not so relevant for decorative hollow products, however, the use of 3D printing for the manufacture of full-bodied structural parts and load-bearing elements of products requires the development of new technological modes that will eliminate warping of parts during printing and allow to obtain a dense homogeneous structure inside and on the structural surface.

To overcome these problems we have developed the DOE plan for future experimental study.

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