Study of polycrystalline alumina materials manufactured by FDM. Statistical correlation of the final properties with the printing parameters.

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INTRODUCTION

Single-phase polycrystalline alumina materials are among the most widely used materials within the family of technical ceramics. These materials are characterized by their high hardness, high resistance to abrasion, high melting temperature and chemical stability [1]. However, their fragility makes it difficult to machine the parts made with them and, therefore, all those forming methods that allow the final geometry and dimensions to be obtained directly are suitable. In this sense, additive manufacturing techniques facilitate obtaining complex geometries. Among them, the FDM (Fused Deposition Materials) technique is still relatively unexplored for obtaining polycrystalline alumina structural materials, despite the fact that this technique is one of the simplest [2].

In this work, alumina pieces have been shaped by FDM for the evaluation, once sintered, of microstructure and bulk density. A statistical study (ANOVA) is used to investigate the influence of the printing parameters on the final properties.

MATERIALS

A thermoplastic composite filament of mean diameter 2.85 mm, containing 52 vol% (83 wt%) of high purity alumina powder with particle size < 1 µm (Zetamix, Nanoe, France) and a polyolefin-based binder system was used for FDM.

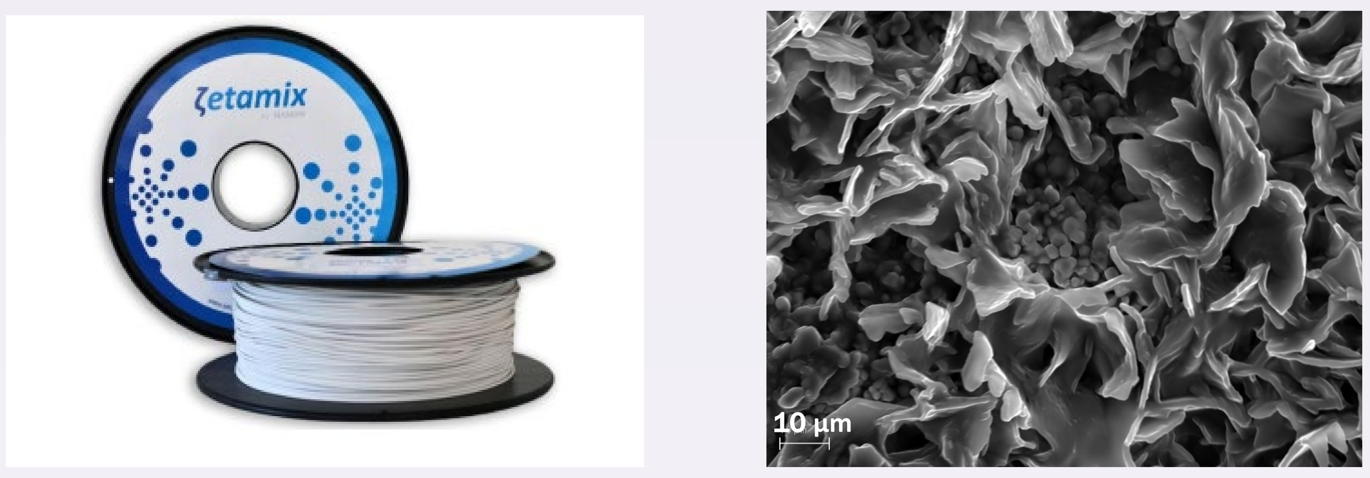


Figure 1. Image of the commercial filament used and detail of the filament fracture observed by SEM

PRINTING PARAMETERS

Cubic specimens of 10 x 10 x 10 mm (after 20% shrinkage in sintering) have been impressed in a comercial desktop 3D printer (Ultimaker S5) with a nozzle of nominal diameter 0.4 mm using the Cura software. For every printing parameter combination at least 5 pieces were printed one by one. Two perimeters created an outer shell and hotend / heated bed temperature was 160/50 ◦C.

Figure 2. Schematic representation of raster angle

Table 1. Main printing parameters

PRINTING SPEED, mm/s	LAYER THICKNESS, mm	RASTER ANGLE
30, 90	0,1, 0,2	0° 0° / 90° +45° / -45°

PROCESSING / CHARACTERIZATION

PRINTED SPECIMENS → CHEMICAL DEBINDING (2 h in acetone bath at 40 °C) → DRYING → THERMAL DEBINDING (8°C/h up to 500°C) → SINTERING (50°C/h up to 1550°C) 2 h Holding time

Materials characterization:

- Bulk Density. Archimedes' method (Standard EN-1389:2003)
- SEM Observations (Merlin, Carl Zeiss)
- Statistical Analysis. ANOVA Test (Minitab® Statistic. Softw.) to find out significance of printing parameters on specimens bulk density.

RESULTS AND CONCLUSIONS

BULK DENSITY

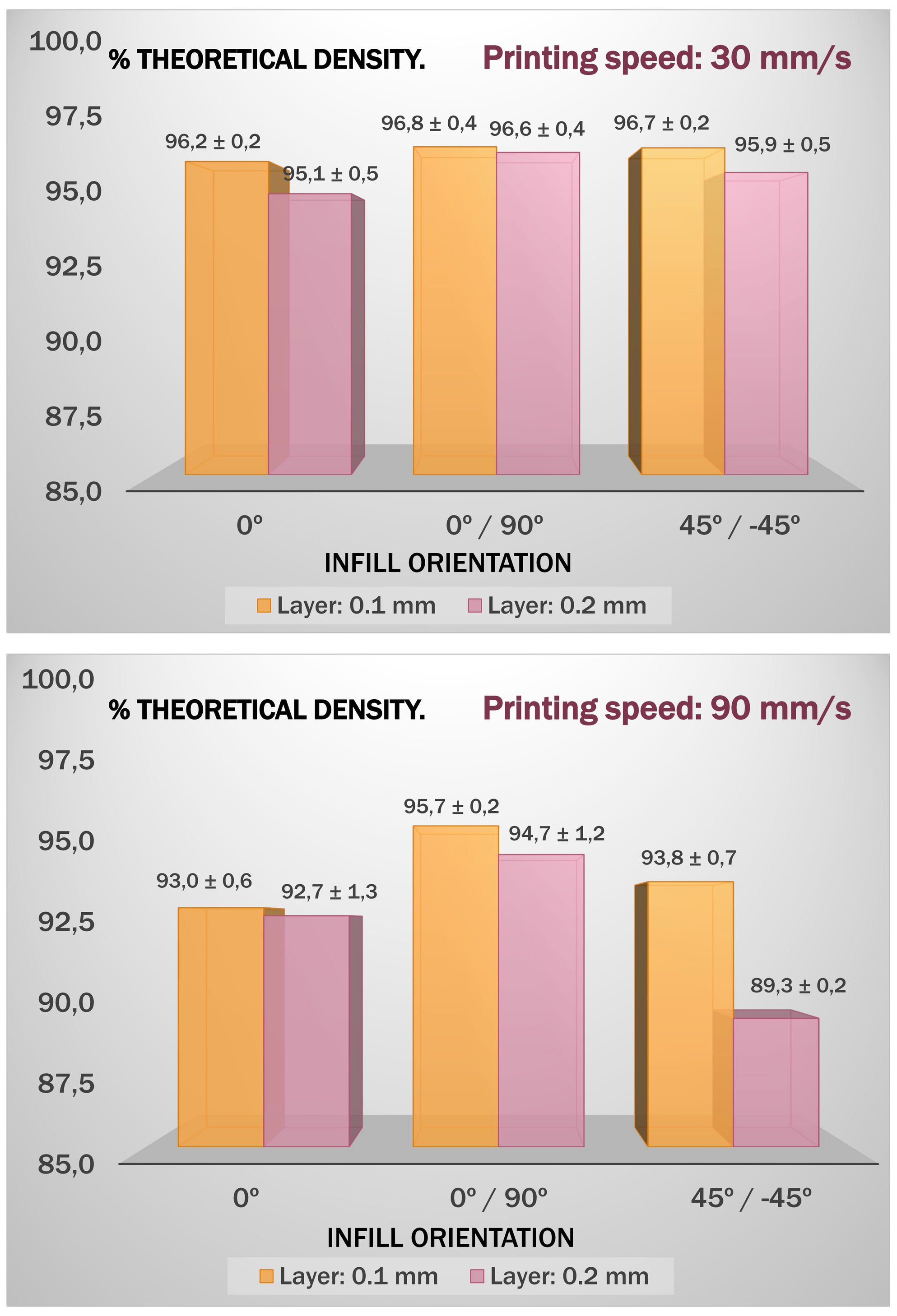


Figure 3. Bulk density expressed as percentage of theoretical density (3.99 g/cm³)

SEM OBSERVATIONS

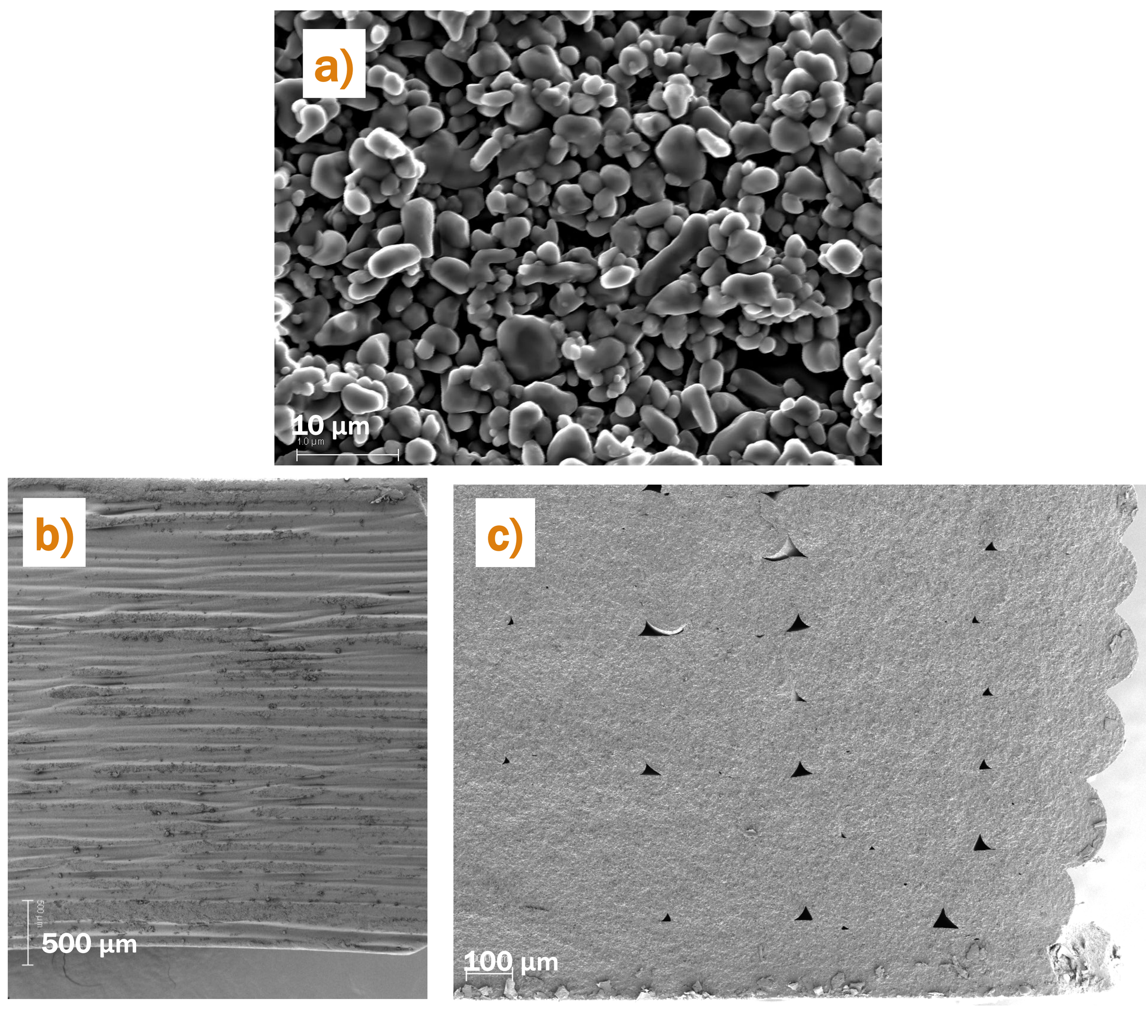
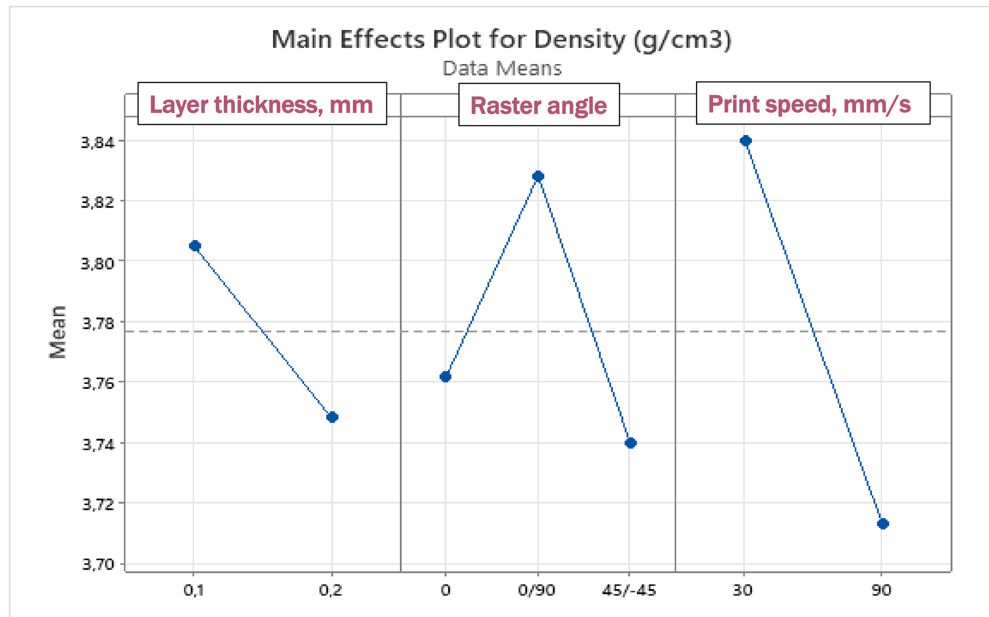


Figure 4. Characteristic SEM observatons of: a) microstructure of green specimens after debinding (prior to sintering), b) layer distribution in sintered specimen, c) flaw distribution pattern along the specimen cross-section

STATISTICAL ANALYSIS



	P-value
Printing speed	0,000
Layer thickness	0,004
Raster angle	0,001

Figure 5. Main Effects Plot for density and p-value to determine whether the differences between densities are statistically significant

- Bulk density values exceed 90% of theoretical one. The printing speed 30 mm/s allows to reach density values higher than 95% of theoretical. On the contrary, the speed 90 mm/s leads to density values between 90 and 95% of theoretical one. Density is greater for a raster angle 0/90 and a layer thickness of 0.1.
- SEM observations allow verifying a good packing of the alumina particles in the green pieces, as well as a relatively good adhesion of the layers in the sintered material. In fracture surfaces, it is possible to detect a characteristic pattern of defects corresponding to the sequence of printing and stacking of the layers.
- The statistical study shows that the p-value is less than a significance level 0.05, so it can be concluded that there are significant differences between the densities achieved with the different printing parameters.

REFERENCES

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