Sterilization study of HP 3D High Reusability PA 12 W

Introduction

This study examines the impact of four different sterilization techniques on parts printed with HP 3D High Reusability (HR) PA 12 W material on the HP Jet Fusion 5420W 3D Printing Solution. The study focuses on changes in color, mechanical properties, dimensional accuracy, and part density. The intent of this document is to provide information about what material properties should be expected after sterilizing parts printed with HP 3D HR PA 12 W.

Test descriptions

We printed samples with HP 3D HR PA 12 W material and investigated four common sterilization techniques for medical devices:

- Steam sterilization: Steam sterilization is performed in an autoclave; it exposes the parts to direct steam contact, at high temperature and pressure, for a specified time period. It is possible to perform several cycles, if a device needs to be re-sterilized after using. We investigated two standard time and temperature settings, comparing the number of cycles as well.
  - One cycle of sterilization at 121°C for 30 minutes, then 30 minutes of drying
  - Three cycles of sterilization at 121°C for 30 minutes, then 30 minutes of drying
  - One cycle of sterilization at 132°C for 4 minutes, then 30 minutes of drying
  - Three cycles of sterilization at 132°C for 4 minutes, then 30 minutes of drying
- Ethylene oxide (EtO) sterilization: Ethylene oxide is a colorless gas used to sterilize devices at relatively low temperatures compared to steam sterilization. For this study, the parts were sterilized using 100% ethylene oxide gas for 6 to 8 hours, at a temperature between 54°C and 60°C.
- Gamma radiations: Gamma radiations are used as a way to sterilize devices because of the ability of the radiations to penetrate products and kill microorganisms. The parts in this study were exposed to radiations between 25 and 40 kGy.
- Electron-beam (E-beam) radiations: E-beam radiations are used based on the same principle as gamma radiations. The parts in this study were exposed to radiations between 25 and 40 kGy.

The color of the samples was measured using illuminant and observer F2/10. Color changes were reported using ΔEcmc. ΔEcmc quantifies the color difference between a target color and a specimen color. A ΔEcmc of 0 means the target color and the specimen color are identical; the greater the ΔEcmc, the greater the color difference between the target and the specimen. In this study, the target color was the color of the sample before the study started; the color of specimens were measured over time and used to calculate ΔEcmc. As a reference, a ΔEcmc of 1 starts to be perceivable by the human eye; a ΔEcmc of 2 is still considered an acceptable color change.

The mechanical properties were measured according to ASTM D638-14, using XY/YX and Z orientated ASTM D638 Type I Tensile Bars per data points. The following data are reported:

- Elongation at break (%), which shows how much the material can stretch before breaking.
- Young's modulus (MPa), also known as modulus of elasticity, which measures the stiffness of the material.
- Tensile strength at break (MPa), which measures the maximum stress a material can withstand before breaking.

The dimensional accuracy of the parts was evaluated by calculating the % of shrinkage from the nominal value on the three different axes: X, Y, and Z. The % of shrinkage was calculated before and after sterilization.

Materials tested and printing conditions.

For this study, a specific plot was designed to reach a 7% packing density using 75% of recycled powder from a previous study and 25% new powder.

Company	Printer used	Material	Powder ratio Used/New	Intended packing density	Build height
HP	5420W	HP 3D HR PA12 W	75%/25%	7%	380 mm

Table 1: Printing conditions.

Results

Summary of data after sterilization.

Although the strength and stiffness of the material are within 5% and 25% change after sterilization, there is a significant increase in elongation at break after sterilization of XY/YX specimens. Changes in color and dimensional accuracy are minimal.

There is an increase in ductility at the expense of stiffness and rigidity.

	% Change elongation at break (XY)	% Change Young modulus (XY)	% Change tensile strength (XY)	Color change	Relative shrinkage	Part density
Ethylene oxide sterilization	+ 56%	- 12%	- 8%	ΔEcmc < 1	< 1%	0.0%
Steam sterilization 121°C 30 min // 1 cycle	+ 91%	- 21%	- 13%	ΔEcmc < 1	< 1%	- 0.3%
Steam sterilization 121°C 30 min // 3 cycles repeated	+ 61%	- 24%	- 15%	ΔEcmc < 2.5	< 1%	- 1.5%
Steam sterilization 132°C 4 min // 1 cycle	+ 49%	- 12%	- 9%	ΔEcmc < 1	< 1%	- 0.2%
Steam sterilization 132°C 4 min // 3 cycles repeated	+ 52%	- 10%	- 7%	ΔEcmc ~ 1	< 1%	- 0.3%
Gamma 25-40 kGy sterilization	+ 36%	- 8%	- 7%	ΔEcmc ~ 1	< 1%	+ 1%
E-beam 25-40 kGy sterilization	+ 50%	- 13%	- 9%	ΔEcmc < 1	< 1%	+ 1%

Table 2: Summary of data after sterilization.

Impact on color

The following graph (Figure 1) shows the ΔEcmc of the top and bottom face of a flat coupon printed horizontally. Most of the values are below 2.5, showing that **the specimen's color changes only minimally during the different sterilization processes.**

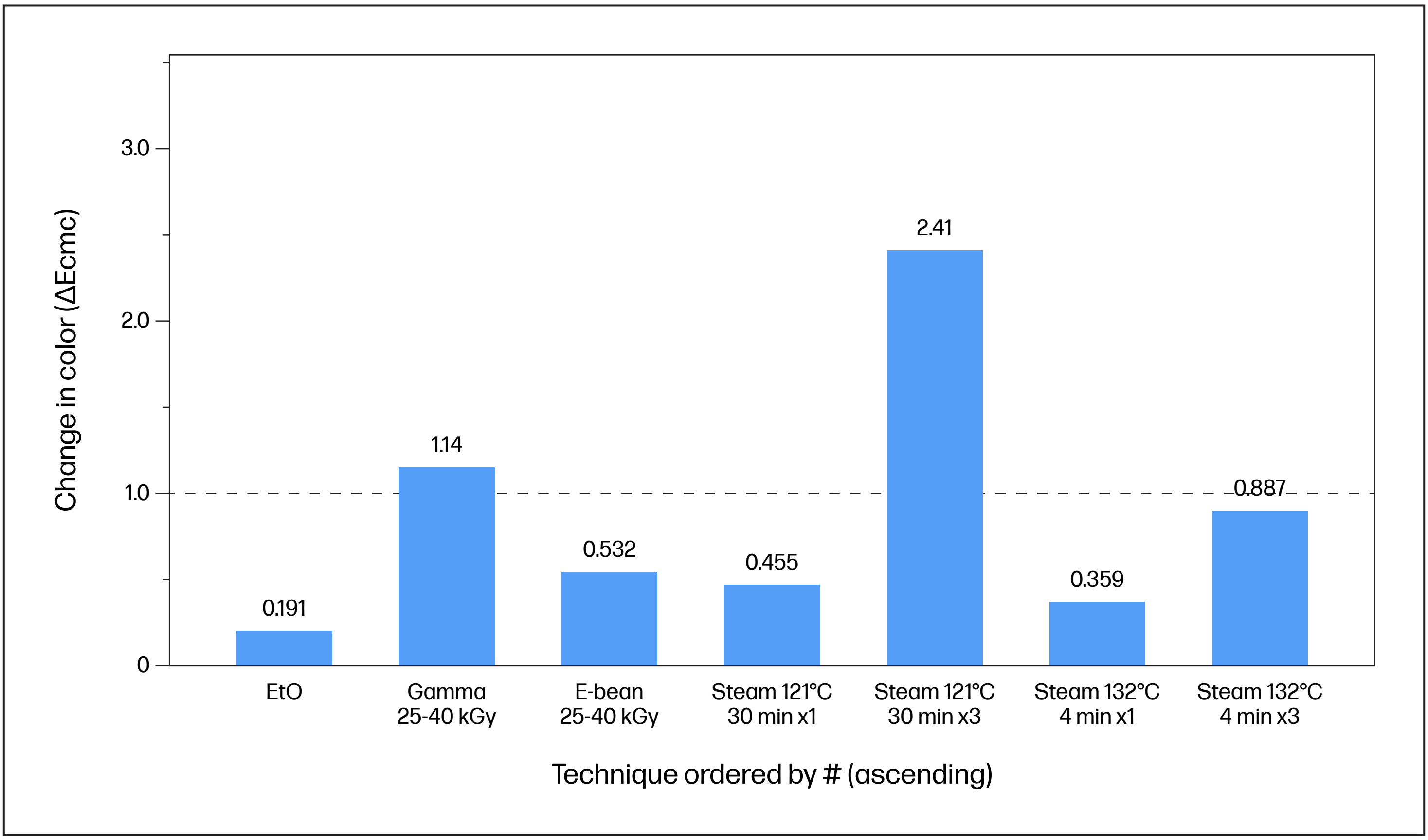

Figure 1: Change in color quantified by ΔEcmc.

The images below show the parts before and after each sterilization process, showing that the impact on color is minimal. We would recommend using deionized water when doing steam sterilization; if tap water is used, water marks may appear on the parts.

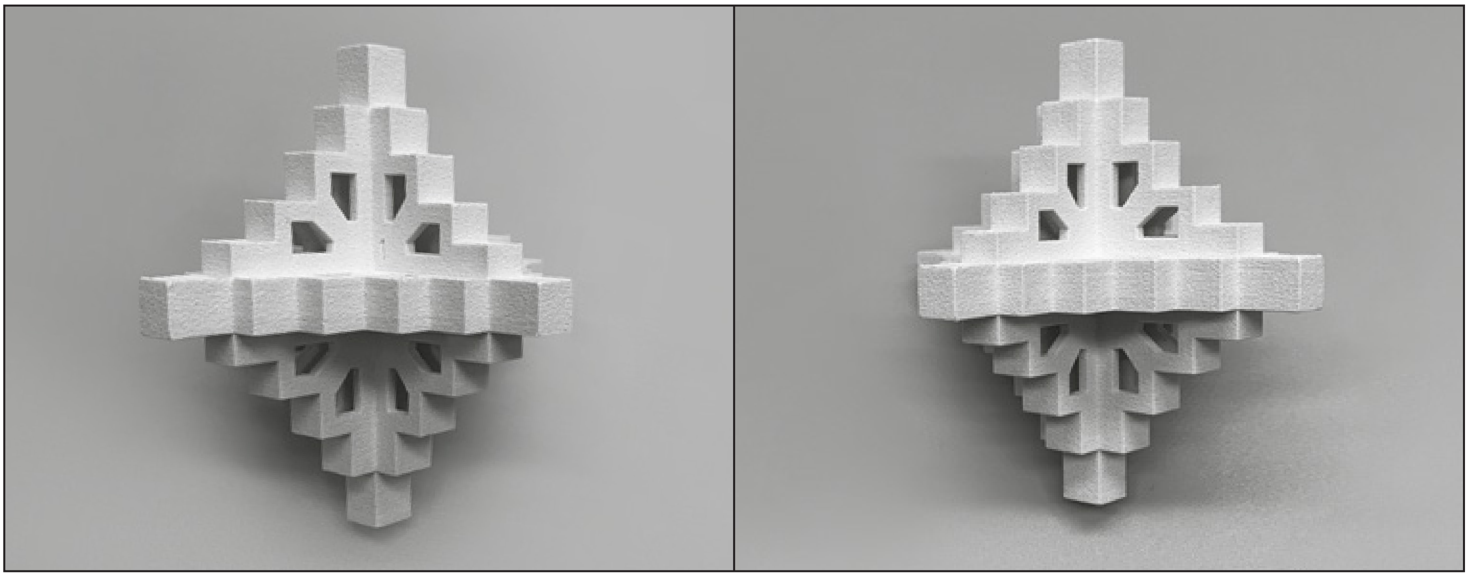

Figure 2: Part before Gamma sterilization on the left, and after Gamma sterilization on the right. No visible change in color.

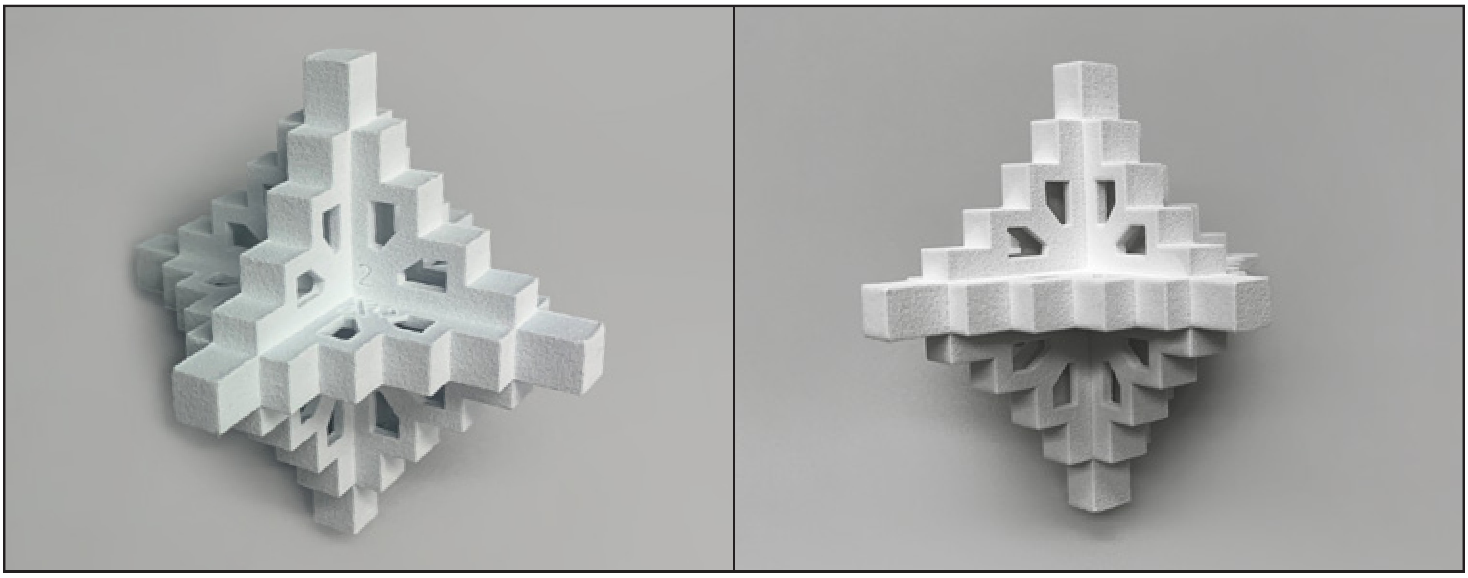

Figure 3: Part before E-Beam sterilization on the left, and after E-beam sterilization on the right. No visible change in color.

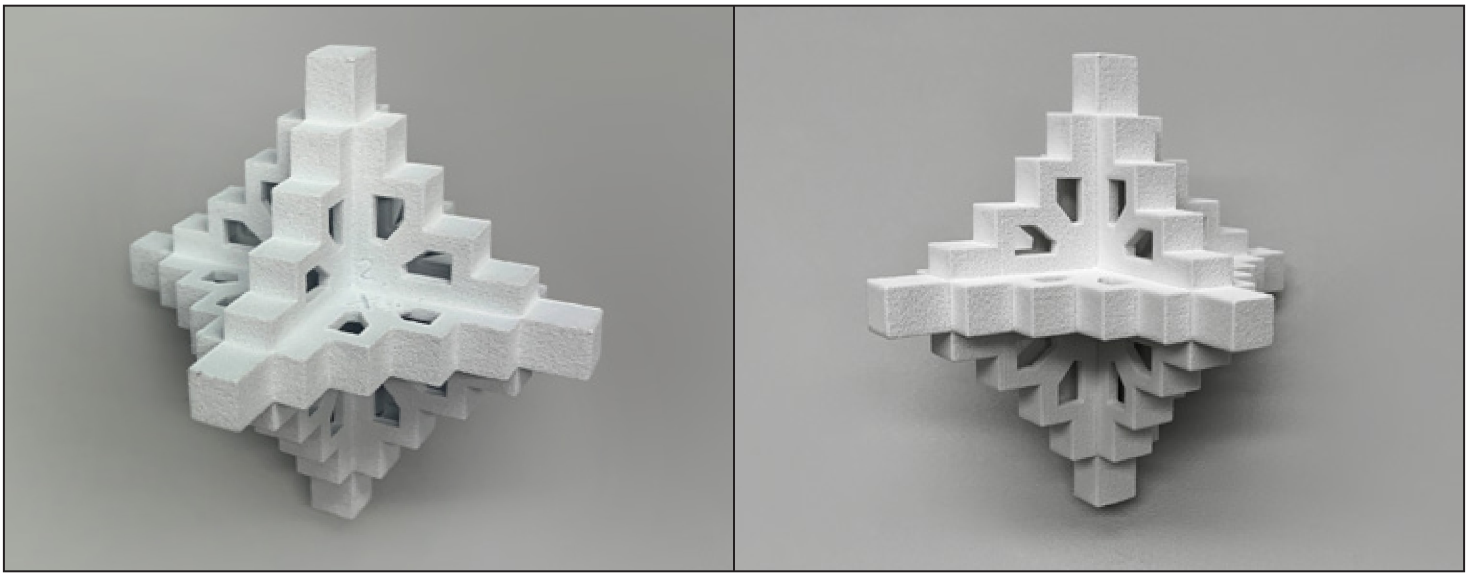

Figure 4: Part before EtO sterilization on the left, and after EtO sterilization on the right. No visible change in color.

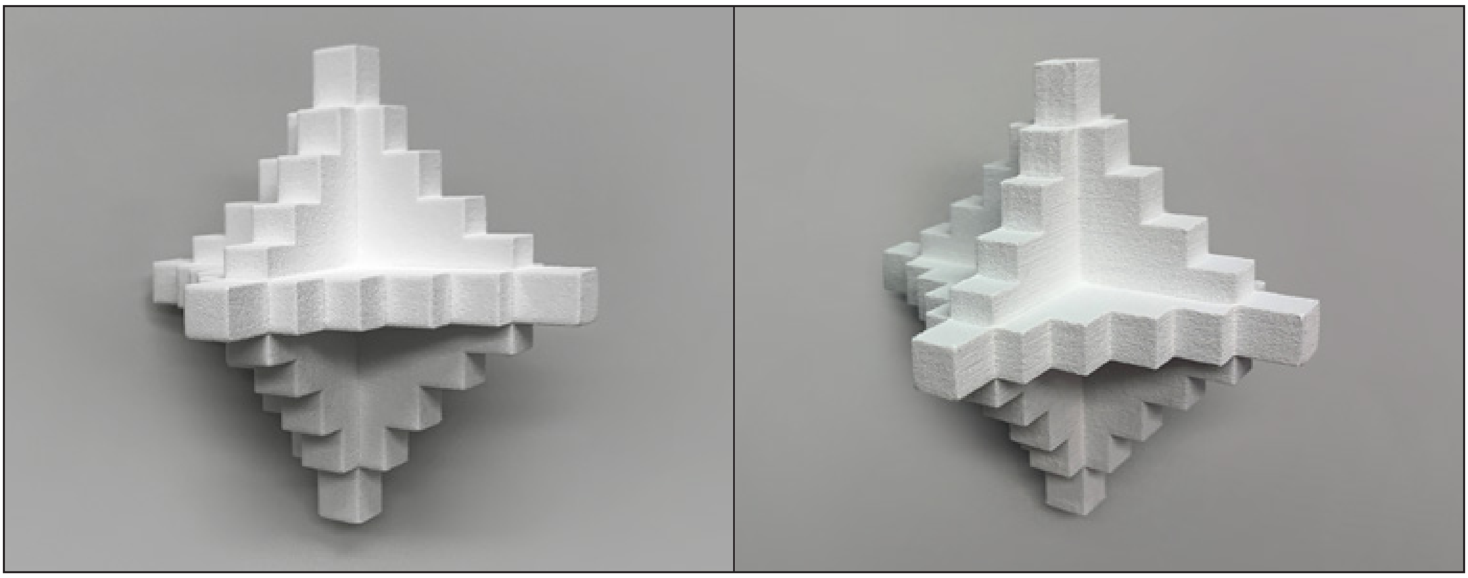

Figure 5: Part after three cycles of before (left) and after (right) steam sterilization at 121°C for 30 min, exhibiting water marks.

Impact on mechanical properties

Most sterilization techniques induce a decrease in Young's modulus and tensile strength, regardless of the printing orientation, while elongation at break in XY/YX is increased after sterilization but slightly decreased in Z orientation.

Impact on mechanical properties is not affected by the type of sterilization.

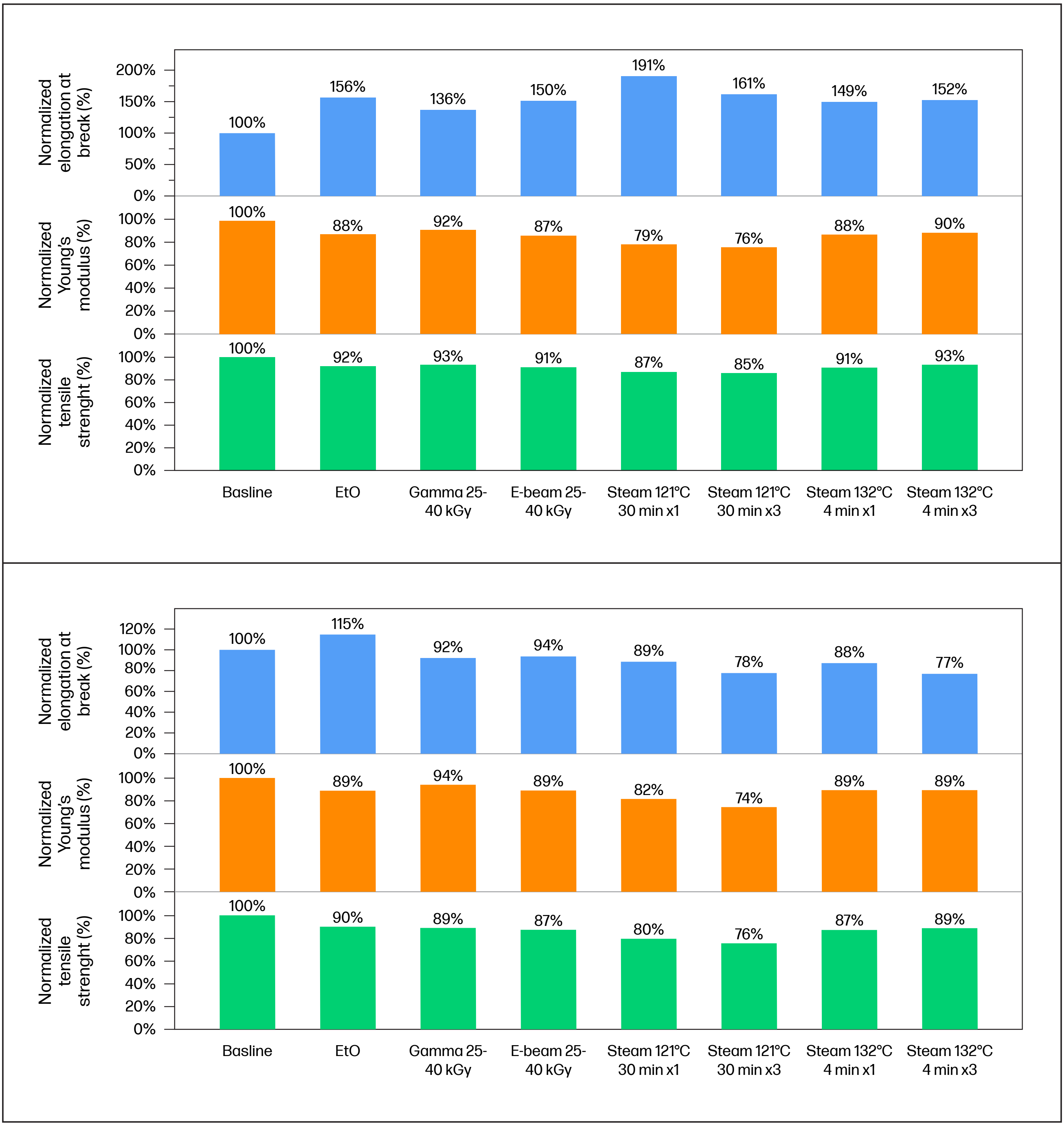

Figure 6: Top XY/YX mechanical properties and bottom Z mechanical properties.

Impact on dimensional accuracy

Dimensional accuracy was evaluated by calculating the relative percentage of shrinkage after sterilization on the three different axes: X, Y, and Z.

There are no significant differences between the percentage of shrinkage before and after sterilization, or between the different processes; dimensional accuracy is minimally impacted by sterilization.

Dimensional accuracy of parts remains stable during sterilization.

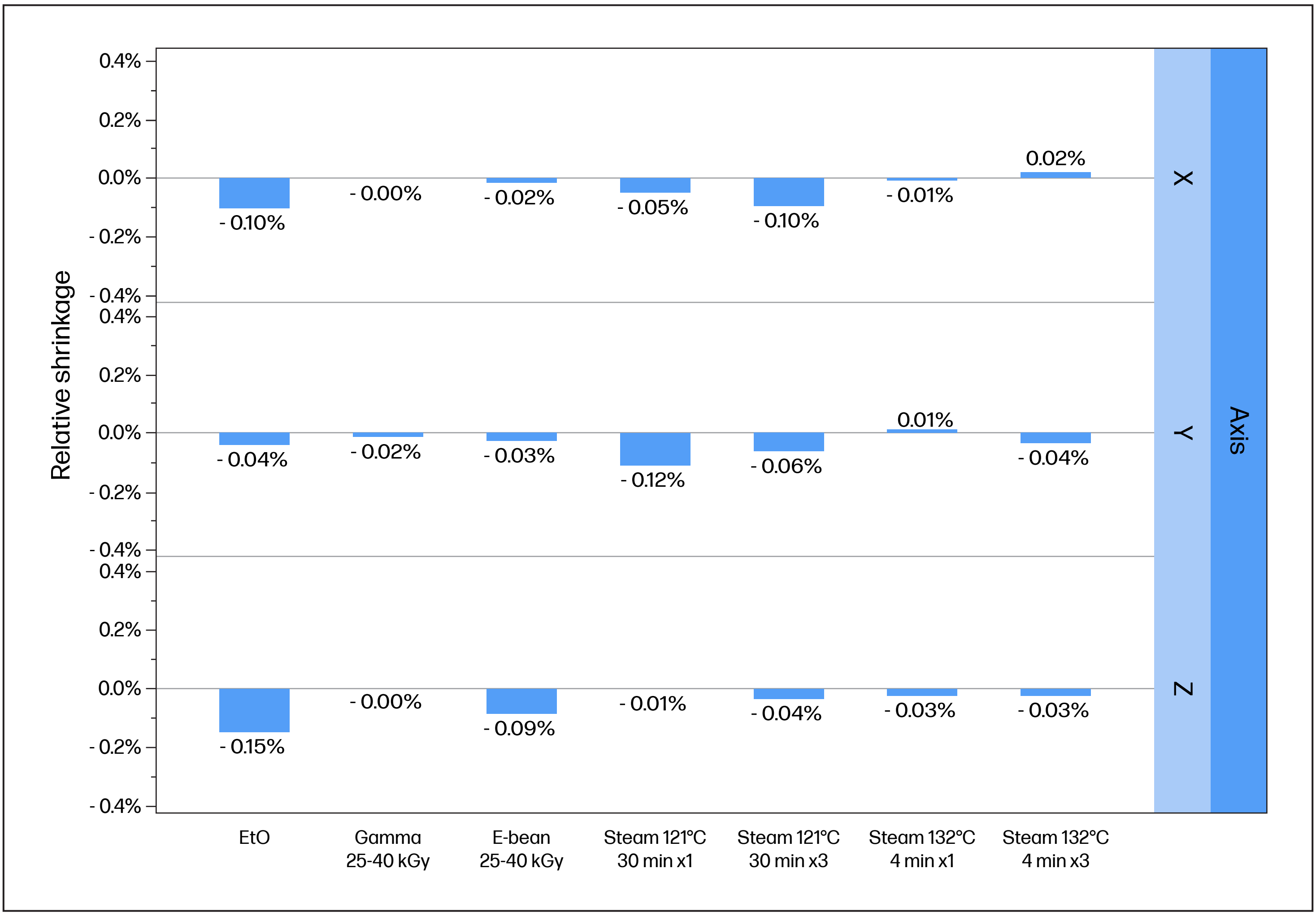

Figure 7: Percentage of shrinkage before and after sterilization is minimal.

Impact on part density

Part densities were measured before and after sterilization; the data are reflected in the following chart. **No significant changes were observed for part density,** meaning the parts can withstand sterilization processes without impact on weight.

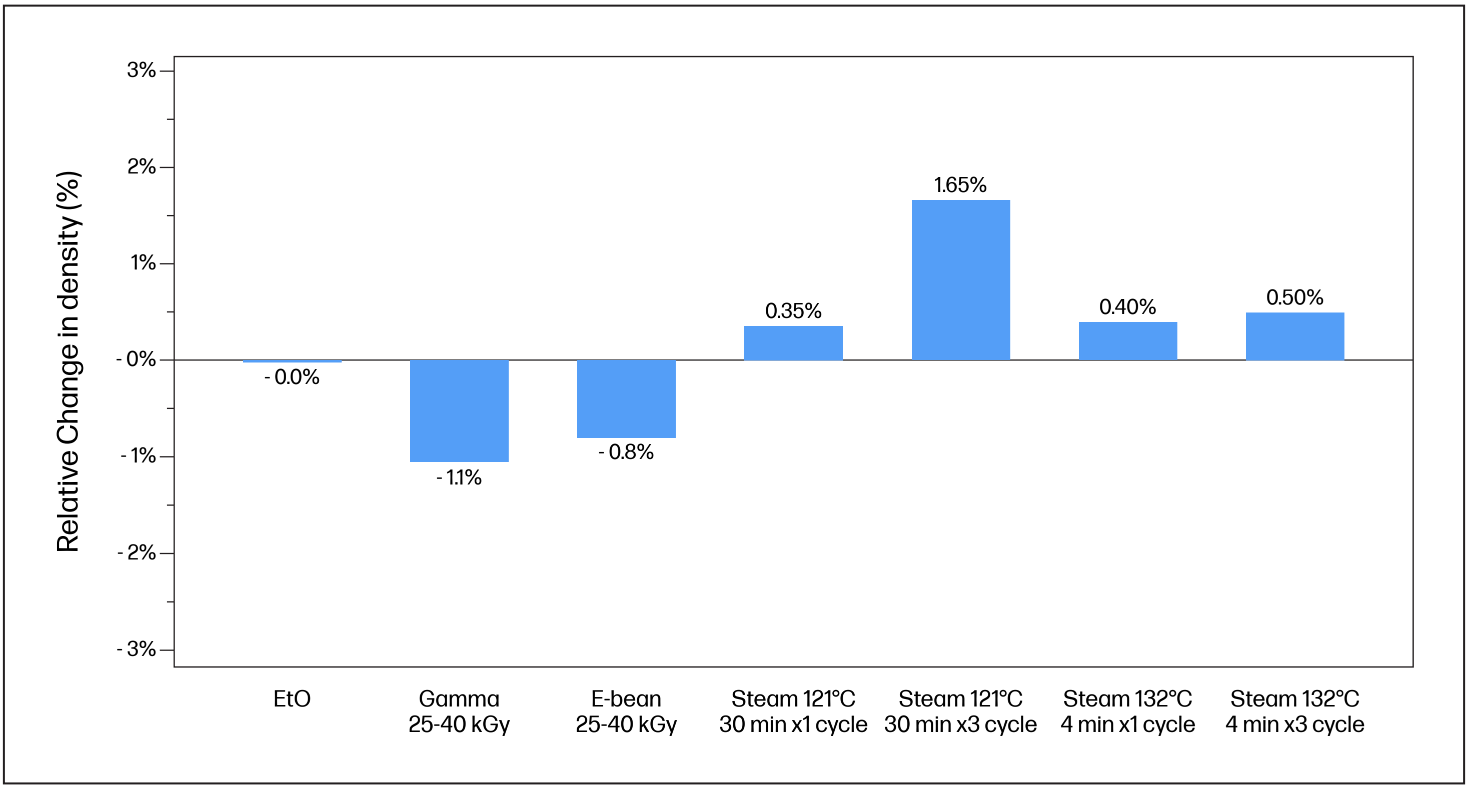

Figure 8: Part density before and after sterilization.

Conclusions

The four sterilization processes studied here reveal similar behavior: an increase in ductility, a slight drop in strength and stiffness, and no change in color and dimension.

Disclaimer: these results are based on test conditions outlined above. They should not be extrapolated.

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