White paper

KEPSTAN®

Oil & Gas seals fabrication via FFF: closing the gap with conventional manufacturing methods

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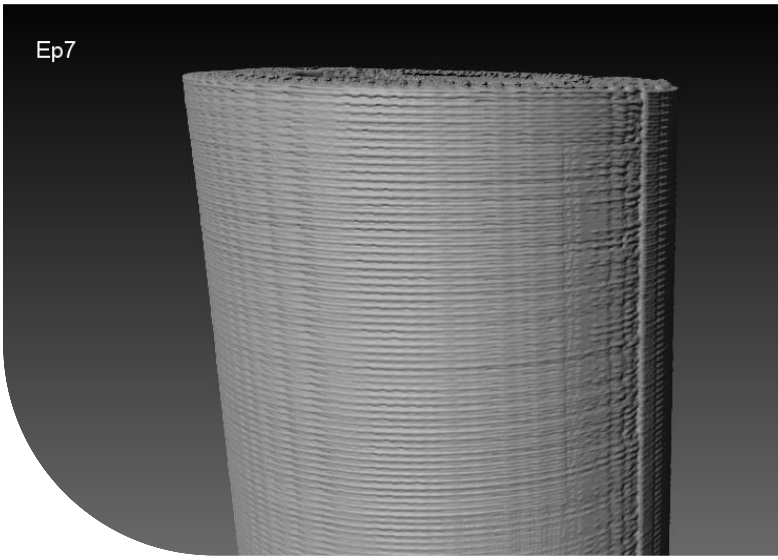
Introduction

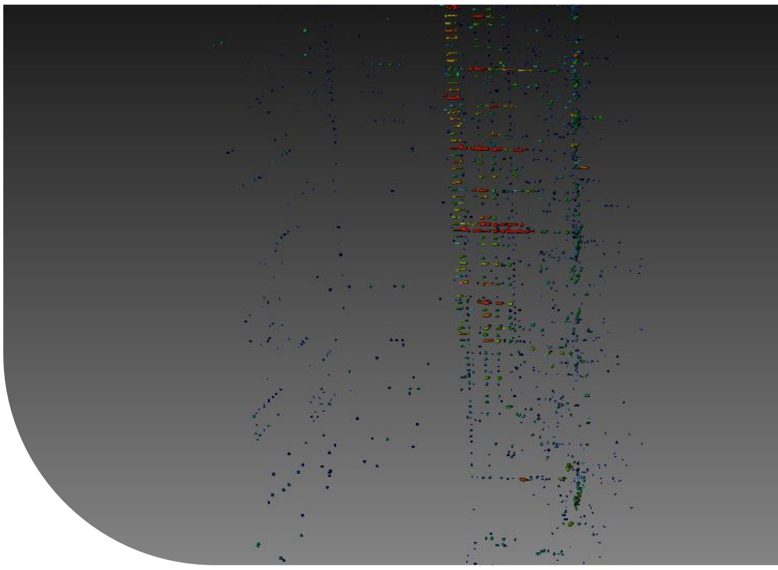
It was previously demonstrated [1] that the mechanical properties of **additively manufactured PEKK** parts nearly matched those of parts obtained by current manufacturing methods (injection molding, extrusion, and compression molding). These technical results firmly established PEKK additive manufacturing as an alternative for producing high performance parts via **a near-net shape approach**. As designers and engineers more widely embrace additive manufacturing, there must be a stronger focus on the economic justification of this new process.

The seals considered here are intended for applications requiring resistance to the most extreme temperatures or aggressive chemicals. In this whitepaper, we address how an increased productivity of the miniFactory Ultra can further make the case of **a viable economic solution** with the Kimya PEKK-SC filament by printing high-quality seals. We first establish the repeatability of the excellent mechanical properties obtained with a standard 0.4 mm nozzle and then move on to a 0.6 mm nozzle, while we show in parallel how using the **larger nozzle can divide the manufacturing costs per printed part by 2.**

Continuous improvement of the PEKK-SC printing process, using a standard 0.4 mm nozzle, resulted in multiple final parts with a density between 1.297 and 1.299. A subsequent post-annealing step, as commonly used in the PAEK stock shapes industry, increased the density to a final value as high as 1.301. These values indicate a theoretical density of more than 99.9 %, which was confirmed by CT-scan analysis, as shown in below images. **The porosity level** was measured as low as 0.02 %. Very low porosity is a necessary condition to obtain good mechanical properties but is also a requirement in O&G applications where RGD (Rapid Gas Decompression) phenomenon can occur.



Tomography analysis showing a porosity level of 0.02 %. Printed part with 0.4 mm nozzle, post-annealed.



Experimental

The material used in all experiments was 1.75 mm diameter **Kimya PEKK-SC filament**, provided courtesy of Kimya (Nantes, FR). Before any printing, the filament was dried at 120°C overnight and kept in the filament drying chamber.

The prints first focused on reproducing the seal geometry used in the first whitepaper [1] (1 cm^2 cross section with an outside diameter of 10 cm), and then considered the case of a ring enlarged by a factor of 1.7 (1.7*1.7 cm^2 cross-section with an outside diameter of 17 cm).

To minimize porosity, a simple toolpath was selected with one outline and alternating layers of perpendicular infill. Both 0.4 mm and 0.6 mm nozzles were used for comparison. All prints used a fixed printing speed of 25 mm/sec and a layer height of roughly 60 % of the nozzle diameter. Flow rates were adjusted to 110% of normal. The same settings were used to print the seal rings and all of the test specimens.

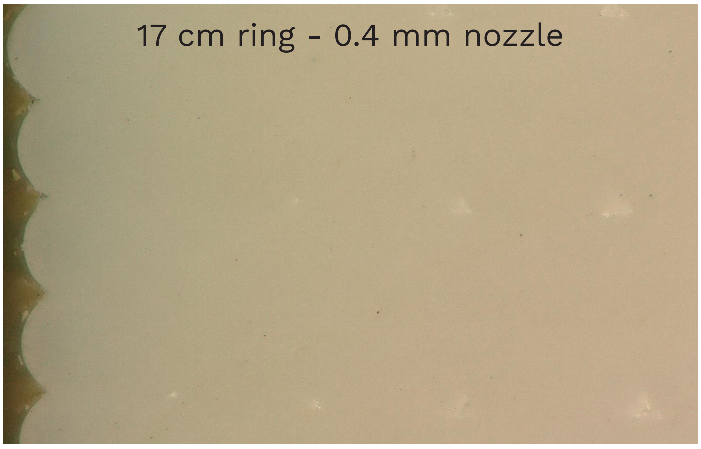
The printing process was performed using miniFactory's **custom process that automatically produces high crystallinity O-ring** without any manual actions from the operator after the initial start of the print. The process uses conditions wherein material remains mostly amorphous until the print is completed. Immediately afterwards, the printer automatically **adjusts the chamber temperature to induce a slow, controlled crystallization.** Aarni, miniFactory's custom process monitoring software, was used to verify that all process parameters stayed within the specified ranges. Full cycle times for the 10 cm rings were about 5.5 and 3.5 hours with the nozzles 0.4 and 0.6 mm, respectively; for the 17 cm rings about 19 and 10 hours with the nozzles 0.4 and 0.6 mm, respectively.

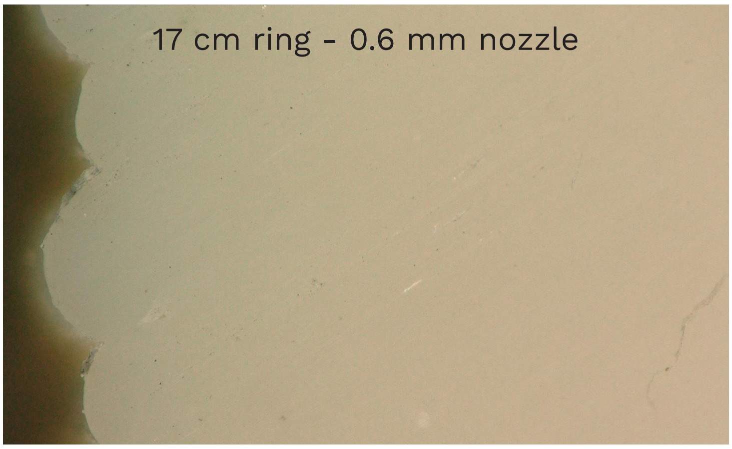
The ring cross-section was checked by Keyence optical microscope at several locations for visible porosity. Crystallinity was measured with wide angle x-ray scattering. In addition to the seal rings, several sets of mechanical test specimens were printed using identical conditions. Tensile XY specimens (ISO 527-1BA 4mm thick) were produced by direct printing. Compression specimens (ISO 604) were cut from the seal rings. All **mechanical tests** were performed without any additional annealing or pre-conditioning.

Results and discussions

The finished rings maintain their shape exceptionally well. Finished dimensions (outside and inside diameter) varied by less than 1% of the original model. Density of the 10 cm and 17 cm rings printed with the 0.6 mm nozzle is ~99% of the typical value for injection molded semi-crystalline PEKK (respectively 1.289 and 1.290 vs 1.303 g/cc). This result is similar to the previous whitepaper, when considering 10 cm ring printed with 0.4 mm nozzle.

Wide angle x-ray diffraction shows crystallinity > 25%. Optical microscopy shows incredibly **low porosity.** Across a number of cross sections there is essentially **no porosity in the bulk**, with only a few small pores where the perimeters meet the infill. In near-net shape processing, this surface porosity would be removed by machining.





Optical microscope cross-section images of the rings zoom x 200

The compressive properties of printed specimens **nearly match an extruded or compression molded PEKK** reference. We observe no difference between the strength and toughness of seals printed by 0.4 or 0.6 mm nozzle. Compressive properties are especially important since it is the main operating mode of a seal.

In tension, the XY specimens show a strength of about 115 MPa, an excellent result matching injection molding properties.

Tensile specimens ISO 527 – 1BA 4 mm thick		Injection molded PEKK	2021 data XY 0.4 mm nozzle	2022 data XY 0.4 mm nozzle	2022 data XY* 0.6 mm nozzle
Modulus	GPa	3.8	3.4	**3.5**	**3.5**
Yield Stress	MPa	110	115	**115**	**113**
Yield Strain	%	5.5	5.5	**5.5**	**5.5**
Strain at break	%	>30	7 - 8	**7.6**	**5.8**

*a large nozzle is not well adapted for the fine features of 1BA test specimens. Shown results come from surfaced specimens to get rid of cracks initiators.

Compression specimens ISO 604		Machined out of Extruded Rods or Compression Molded Plates PEKK	2021 data Machined out of 10 cm printed ring 0.4 mm nozzle	2022 data Machined out of 10 cm printed ring 0.6 mm nozzle	2022 data Machined out of 17 cm printed ring 0.6 mm nozzle
Modulus	GPa	3.9 - 4.2	3.5	**3**	**3**
Yield Stress	MPa	165 - 175	150	**149**	**150**
Yield Strain	%	6.5	7 - 8	**8.1**	**7.6**

Conclusion

In this whitepaper, rings of 2 different dimensions were **printed with a larger nozzle than standard** (0.6 mm versus 0.4 mm) on miniFactory Ultra system with Kimya PEKK-SC filament. With such higher throughput, the total process time for bulkier parts -such as the 17 cm rings- was reduced from 19 to 10 hours. Including machine warm-up, printing, part annealing and cooling, this is a reduction of 47 %, which directly **translates to theoretically twice as many parts produced in the same total number of operating hours** per year for one system.

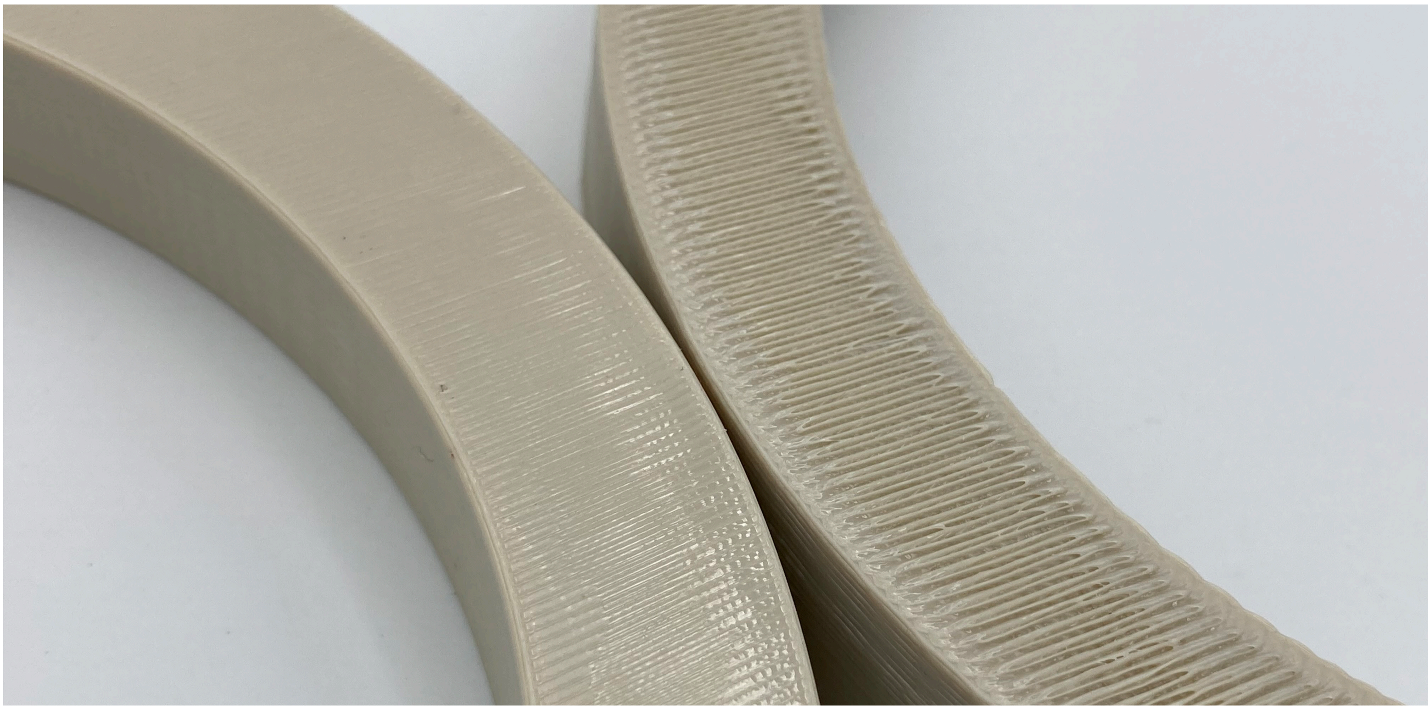
At the same time, we showed that similar high mechanical properties could be obtained by using the 0.6 mm nozzle as compared to the standard 0.4 mm nozzle. In particular, the behavior in compression - the main mechanical stress experienced by seals - is remarkable.

Outlook

The collaborative work between **Arkema** and **miniFactory** shows that is possible to produce at an increased productivity Kepstan® PEKK near net shapes seals displaying excellent mechanical properties.

Thus, a full technology package (miniFactory Ultra, Kimya PEKK-SC Filament, Arkema Kepstan® PEKK) is now in place to challenge PAEK stock shapes machining, the current mainstream method to produce these seals.

Arkema, miniFactory and Kimya are currently working with end users to further leverage the benefits of additive manufacturing vs. machining, in particular in terms of **inventory reduction** (“One Filament, Many Seals”) or increasingly in terms of waste reduction.



Left: 0.4mm nozzle
Right: 0.6mm nozzle

References

(1) Xavier BERGAMINI, Philippe BUSSI, Olli PIHLAJAMÄKI, Riku HIETARINTA. “Semi-crystalline Kepstan® PEKK Seals via Fused Filament Fabrication on the miniFactory Ultra®”. 2021. https://www.arkema.com/global/en/resources/post/webzine-post-white-paper-semi-crystalline-PEKK-seals/

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