How the ESRF is Using Zetamix for High-Temperature Sample Environment

A synchrotron is a particles accelerator that generates one of the world's most intense X-ray source, 100 billion time brighter than X-ray used in hospitals. Thanks to this technology, scientists are able to analyse the structure of matter down to the atomic level. Located in Grenoble, France, the ESRF is the result of the cooperation of 22 nations. Each year, more than 9000 scientists come to ESRF and use one of the 43 beam lights for their experiment. Most of these experiments require specific sample environment (cryogenic, high temperature, in situ chemical reactions) and high reactivity from the staff while experiments are running (as beam time is precious and allocated months or year in advance). This combination explains the need for custom parts made in technical material in less than a few days at the ESRF.

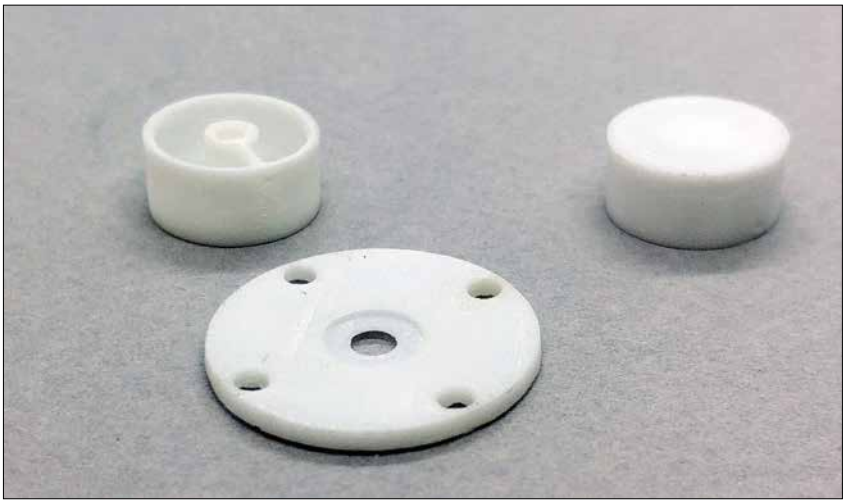

Fig. 1
Two heating cartridge shells and a sample holder printed in Zetamix

Work at ESRF

Carlos Cosculuella and Yves Watier work for the Samples Environment Service and are specialised on extreme environment. Their daily mission is setting up equipment adapted for experiments on very low or high temperature environment. Not also their presences are crucial to troubleshoot scientists with deficient devices but, above all, to create new instrumentation and improve the quality of experiment.

Keywords
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C. Cosculuella and Y. Watier are designing prototypes and on demand pieces every day. Finding an industrial partner able to produce small quantities, even for plastic parts, is complicated. Zetamix technology and more extensively 3D-printing provides them autonomy and versatility: It is the easiest way to produce sample holders, heating elements, gas blowers components and other parts on demand. Already equipped with Prusa 3D-printer for prototyping in plastic, C. Cosculuella and Y. Watier account among our earliest customers to use Zetamix, the first ceramic and metal filaments suitable with FFF 3D-printers.

Zetamix technology: the quickest way to produce ceramics simple parts

Zetamix filaments are convenient to produce ceramic samples in a record time.

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"Some scientists planned one week of experiment one year in advance. Their time on the synchrotron is precious, they can't afford wasting time with equipment troubles" said Y. Watier.

For example, Yves was solicited once by a scientist who needed to hold his sample on a heating resistance. The experiment was already ongoing, but the initial sample holder, made of stainless steel, was too much heat conductive, and thus, the sample was not at the targeted temperature (Fig. 2–3). The part requested was very simple, basically a flat washer in ceramic with five holes and a chamfer, but impossible to get in ceramic in less than one month.

"The scientist needed this part as fast as possible, in order to save his allocated beam time. Thanks to Zetamix, we designed, printed and debinded it the day he asks us for help, we sintered it during the night and made our test the day after. 32 h after his request, the sample holder was ready to use" (Fig. 2).

Making the object by traditional way would be costly and above all would have taken too much time. "That kind of part are actually too simples. Most cases, we can't even get quotes for these parts, and when we do, it would take two to four weeks to get the quote, and then another month to get the part" said C. Coscuella. To answers this kind of issues, the of predecessor C. Cosculuella and Y. Watier used to buy a batch of standard alumina parts, and they would adapt the experiment around these, with a bit of drilling, polishing , etc. "There was an evolution in the way the issue was considered, now we are looking for part which are adapted better to the experiment. Finding a partnership with ceramic manufacturer would be too long, expensive and complicated, Zetamix was the best solution" said Y. Watier.

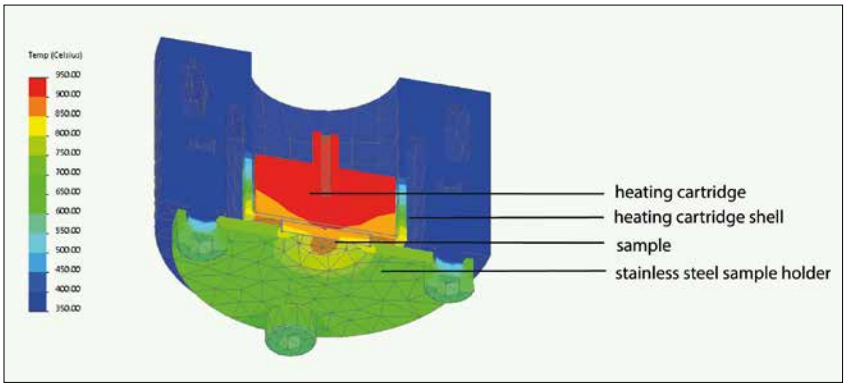

Fig. 2
Solidworks thermal simulation with inox sample holder

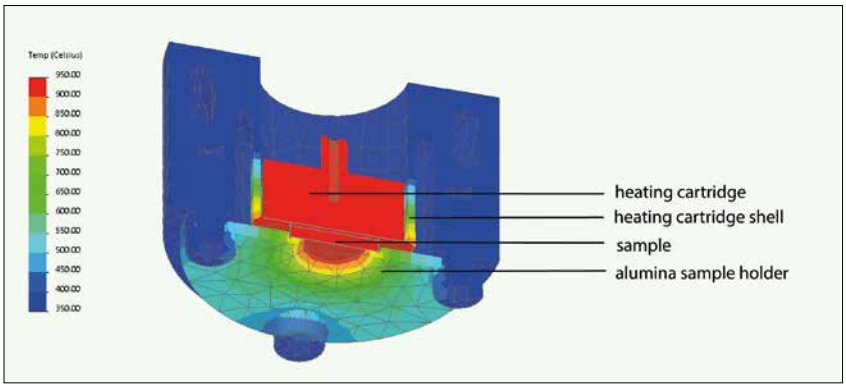

Fig. 3
Solidworks thermal simulation with alumina sample holder

Zetamix technology:
the solution for custom made parts

For many experiments, the ESRF needs to use a sample carrier equipped with resistance, called heating cartridge. Standard products for this need are quite expensive, and only in standard sizes and power. C. Cosculuella and Y. Watier needed tailor made heating cartridges, and haven't other choice than making them by themselves.

Before using Zetamix technology, the process of fabrication of this part was pretty complicated. C. Cosculuella and Y. Watier used to cast a refractory cement in a plastic mold to trap the resistance. However, the resistance was likely to move while the cement was drying, triggering default on the sample carrier that make it unsuitable or dangerous to use. When the plastic mould

Tab. 1
Zetamix vs. conventional process

	Outsourced Ceramic	Standard Part	Zetamix
Properties	Heat insulator	Heat conductive	Heat insulator
Price	200 EUR	20 EUR	5 EUR
Lead time	1–2 months	1 week	32 h

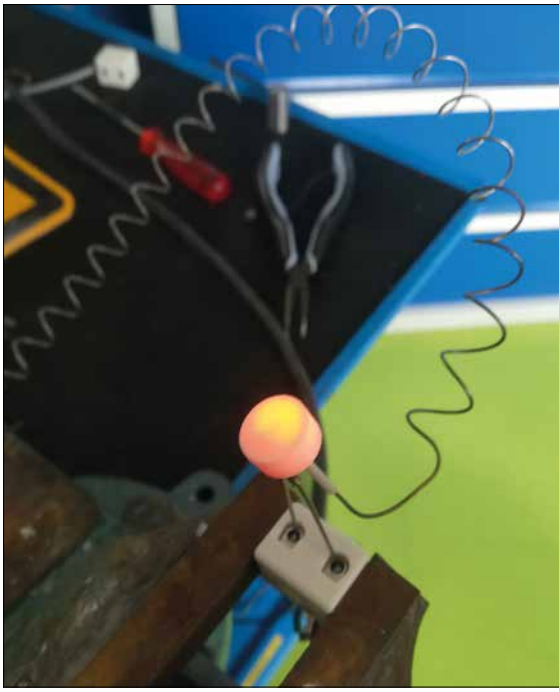

Fig. 4
Heating cartridge printed by Zetamix and assembled with heating element

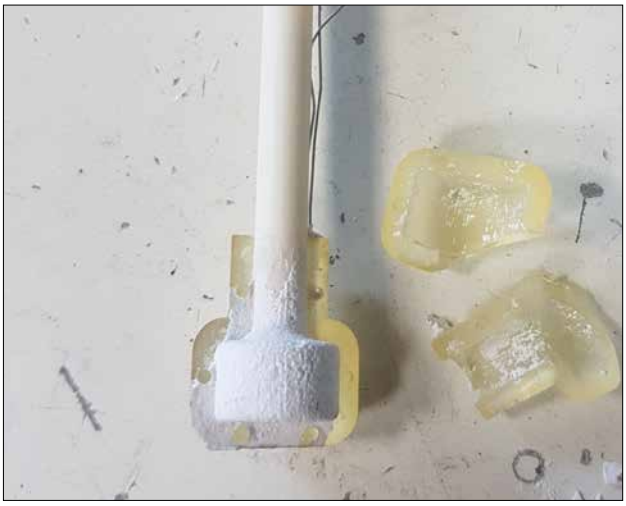

Fig. 5 a–b
Heating cartridge assembled by hand vs. printed by Zetamix

Tab. 2
Zetamix vs. standard and manual assembly for heating cartridges

	Outsourced	Manual Assembly	Zetamix
Shape	Standard shapes	On demand shape, poor quality	On demand shape, perfect quality
Process	outsourced	Complex process	Easy to produce
Price	50–200 EUR	10 EUR	10 EUR
Lead time	1 month	1 week	1 week

was removed, some parts of the resistance could outreach the shape on cement. This difference of environment made a stress on the resistance that could shorten its time of use, and could generate some sparks. Not only this process was long, but it could fail and the final cartridge had a limited time of use (Fig. 5 a–b).

Thanks to Zetamix, they easily succeeded to cope with this problem. They have designed a shell that perfectly fitted the resistance shape, and instead of adding a divider to separate the different electrical connections, they have modelized it with the rest of the object. When the cement casted, the resistance cannot move and they don't have to remove the mould, which is now part of the final cartridge. Zetamix also allows for a very wide range of sizes, shapes, in and out electrical connections, holding fixtures.

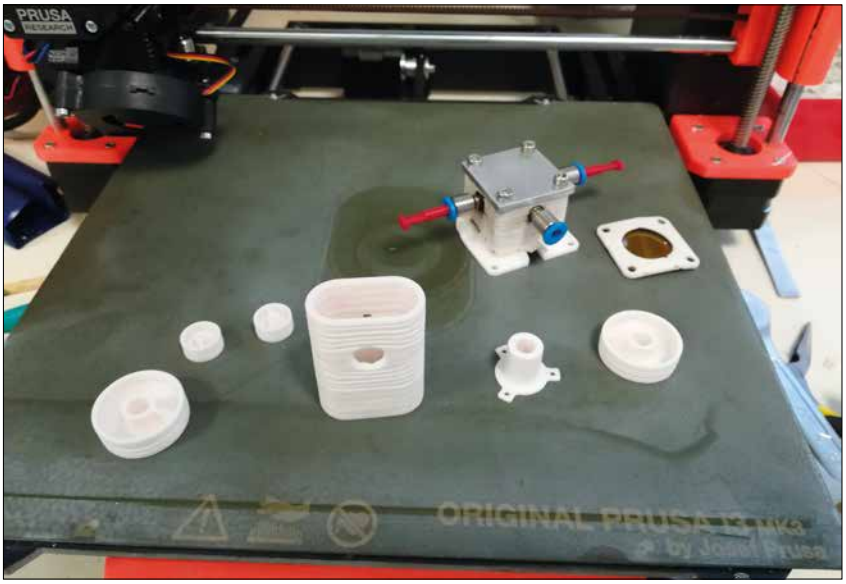

Fig. 6
Different parts printed in Zetamix: three different sizes of heating cartridges, two sample holders, and a prototype of micro-furnace

Zetamix technology: a wide range of possible applications

In addition to being time and money saving, Zetamix filaments user gain access to a remarkable versatility. Not only it allows C. Cosculuella and Y. Watier to improve their regular equipment but it is also an opportunity to try new designs and to create new sample environment, much more adapted to scientists' use.

"We are working at the moment on much more complex designs, for full environment control using Zetamix. For instance, we have the project to produce a water-cooled micro furnace, with inner channels for cooling, reactants and temperature measurement. That kind of part is not ready yet, but when it will be, it will improve greatly the quality of analysis for some specific experiments. And of course, this could not be done with any other technology."