How to grow new crystals for high-tech applications

High quality single crystalline materials are at the heart of many high-tech applications, from high-power lasers to the rare-event detectors essential for the discovery of new particles. However, despite the commercial and research need for such materials, growing them reproducibly to the required standards is not an easy task, but one that Dr Matias Velázquez at the Institute of Chemistry of Condensed Matter of Bordeaux, and Dr Philippe Veber at the Institut Lumière Matière in Lyon, France, have found a way to overcome. By developing new methods for crystal growth, their teams have been able to grow crystals for several commercial applications.

Cryostats are a special kind of solid where the atoms are arranged in a highly ordered, repeating 3D structure. Precious stones such as rubies and sapphires are examples of naturally-occurring crystals, formed when small particles escape from hot magma inside the Earth and are squeezed through narrow fractures and cavities, where the particles can cool and grow in a process called crystallisation.

The highly-ordered atomic structure of crystals gives them many unique properties that differ from less-ordered solids and is key to many of their applications. Crystals are commonly found at the heart of laser systems, where different elements can be substituted in the crystal structure to produce different colours of laser light. They are also key in many other technologies, such as other laser optics, specific types of optical insulators and the direct detection of particles in high-tech applications.

Most of the crystals in these high-tech applications are synthetically grown by controlled and engineered directionnal crystallisation. Reproducibly growing high-quality crystals is a fiendishly difficult task. Many lab-grown crystals are normally made from cooling super-saturated solutions of the chemicals of interest with a growing platform, like a wire, in the solution. As the solution cools, the solubility of the chemicals decreases, and they begin to solidify on the wire, forming the crystal structures. However, there are many factors that influence the size and quality of the final crystals, from the rate of cooling to the presence of dust or other particles in the solution, making it difficult to fully control the process.

Doctors Matias Velázquez and Philippe Veber may have an answer to the problem of how to grow high-quality crystals reliably. Their research focuses on developing methods to grow single crystalline materials, a type of material which has such a regular structure that it can be considered as one bulk single grain, offering the possibility to exploit their intrinsic physical properties as well as their anisotropy. This means that the crystal needs to be as free as possible of even the smallest defects. Much of their work has also been carried out with several international collaborations, including a particularly productive one for growing crystals mainly for optical applications with Dr Daniel Rytz, physicist and R&D director at FEE GmbH, Germany.

The flux technique that Velázquez and Veber developed led to hitherto unknown crystalline materials such as cubic terbia and heavily substituted gadolinia oxides. While the latter was proved to lase, a promisingly high Faraday rotation was discovered in the former, opening new perspectives in optical insulation and polarisation circulation in high power laser devices. We speak about the same kind of flux growth technique that such brilliant companies as FEE GmbH or Cristal Laser S.A. have turned into a ton-scale production industrial process, developing special crystal structures that have not just found many uses on Earth, but some have made their way to Mars.

SINGLE CRYSTAL SESQUIOXIDES

Velázquez and Veber have particular expertise in growing single crystals of cubic rare earth sesquioxides. Sesquioxides are defined by having a ratio of three oxygen atoms for every two metal atoms and most of the crystals used in laser applications fall into this category of refractory materials. Changing the combination of the rare earth metals in the sesquioxides and the metals used as dopants mean that these sesquioxides are used not just in high-power lasers, but also eyesafe lasers for telecommunication, scintillator materials for x-ray materials, upconversion materials for next generation of solar cells or Faraday rotators in optical insulation.

The method Velázquez and Veber have somewhat renewed is known as the flux method. This is a cheap, simple, reliable method that uses a non-toxic solvent in which the crystals grow at half of their melting temperature, significantly lower than other growth techniques. The growing chamber for the crystals can be run in air but one of the key components that has made this technique so successful for growing is the discovery of a solvent family that is particularly effective at favouring the desired crystallisation patterns while incorporating many of the rare earth metal ions. The growing process can also be carried out in air and should be capable of producing hundreds of single crystals pieces demanded by the highly selective niche markets, and also allows Velázquez and Veber to be the only one in the world, even nine years after achieving their world premiere in the synthesis of new crystal types, to be able to obtain these efficient materials.

Being able to produce such quantities of crystals and single crystals of large sizes is ideal for laser applications. Another of the unique features of the cubic rare earth sesquioxide single crystals is their ability to dissipate isotropically heat and deal with the high-thermal load conditions in high power laser systems. The properties of the crystals grown by Velázquez and Veber compare favourably in terms of efficiency and wavelength tunability to those grown by traditional methods, but their approach is not just more efficient in terms of energy and cost but offers greater flexibility in terms of the metals that can be introduced uniformly to the crystals. Crystals can literally be tailor-made. This is part of why this synthesis method has already found several commercial applications.

If some of these crystals – like the flux grown RTP ones by Cristal Laser S. A.– have made their way to Mars, we should be able to reach the markets.
PARTICLE DETECTORS
Being able to grow crystals of large sizes on larger scales has proved very useful for rare event detectors. These are the detectors used at places like CERN to record data on the particles formed from high-energy particle collision experiments. Another example is the CUORE detector, located far underground in Italy, which looks for very rare events in baryonic matter likely to radically change our views on the ultimate components of matter.

The scale of both detectors and the number of crystals required for them is colossal. At CERN, there are over 90 tons of crystals, with over 60,000 crystals located in the detector barrels alone. The CUORE detector is nearly 19 stories tall and contains nearly a thousand crystals as well. But current crystals implemented at the core of this detector are not scintillating, and Velázquez and Veber have decided to take up the challenge of producing the next generation of crystals for heat-scintillation cryogenic bolometers.

Incredibly low levels of background noise is essential for achieving detectors with extremely high levels of certainty required for detection of these very rare decays. The production of such materials has not just involved trial and error in the laboratory but also the integration of numerical simulations to guide the design process.

OUTLOOK
Velázquez and Veber will not just stop at these applications. Their growth methods can be adapted for composite crystals, different types of crystals that are joined together, that are of interest in laser applications and specially shaped crystals that can be used to make very hard, durable windows. Their work has already found industrial interest, but their versatile, reliable growth techniques are likely to find more applications in the future, including continuing to increase the amount of rare earth substitution in the crystals, as Velázquez and Veber believe they have yet to reach the limits of their synthetic methods.

CONTROLLING CRYSTAL GROWTH

Controlling crystal growth and reproducibly growing high-quality and yet unknown crystals is a fiendishly difficult task... one that Drs Velázquez and Veber may have an answer for.