2theta

MAGAZINE

TAKING THE PULSE OF MARINE ECOSYSTEMS

p. 6

VISUALIZING STRUCTURAL DiverCT

p. 10

THE STRESSES OF AIRCRAFT SAFETY

p. 14

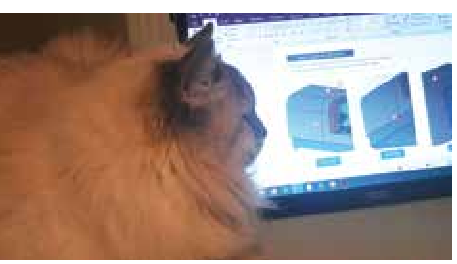
SPRING 2020 / ISSUE 3

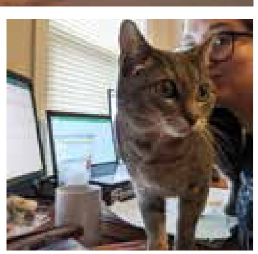
FROM THE TEAM

a message for our readers

As a result of the COVID-19 pandemic, we've all had to make significant changes to our daily lives and adapt to new rules. The Proto team is grateful to have the tools and technology to stay safe while working and supporting our customers during these difficult times. Despite the challenges, one thing is certain: our sense of community has become stronger than ever. Within the scientific community specifically, we've witnessed researchers taking to Twitter and other online platforms to share their work and make up for lost conference time.

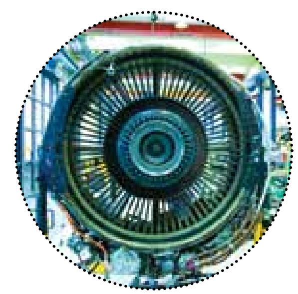
While we all do our best to adjust to the new normal, we hope that you can take a moment to appreciate the stories of scientific innovation we present in our articles. Although we haven't been able to connect in person, we can always learn something new, strike up a conversation, and bond over science. We extend a heartfelt thank you to our readers for their support so far, and we welcome you to share any ideas you may have by emailing us at 2theta@protoxrd.com.





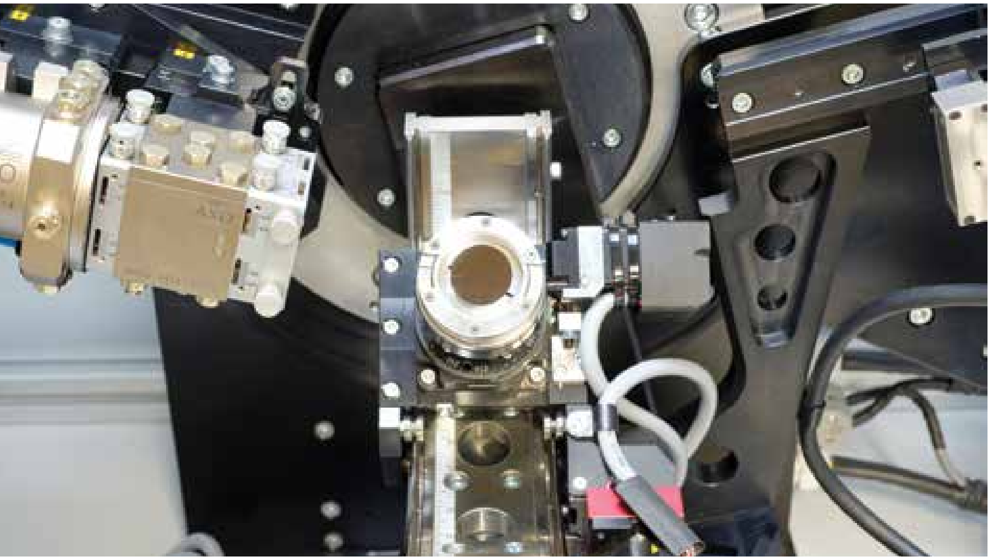
2theta MAGAZINE

A magazine highlighting x-ray innovation in science and engineering.



THE SCOOP

what's happening around Proto



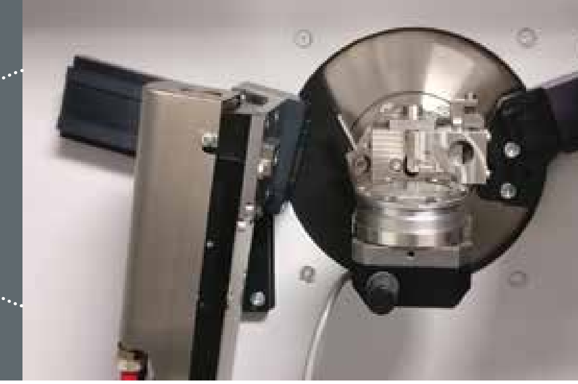
Although we can't possibly detail all of Proto's developments over the past year, we are excited to share a brief highlight of our growth in 2019. The Proto team installed numerous systems at a variety of institutions, including an AXRD Benchtop at the Federal University of Rio de Janeiro (Brazil), a Laue-COS at the University of Stavanger (Norway), and an LXRD Microarea at Dalhousie University (Canada). In addition, we expanded our accessory offerings for our AXRD LPD and AXRD HR systems: they can now be equipped with a Eulerian cradle to facilitate stress and texture analysis, a 2D detector to provide information about preferred orientation and strain, and x-ray reflectometry tables to allow users to orient samples horizontally to the x-ray beam. Lastly, we are proud to announce that after years of experience in x-ray tube manufacturing, we have expanded our in-house x-ray tube facility to meet increasing production demands. Thanks to this ISO 9001 certified facility, we have been able to triple our previous x-ray tube production capacity. Our team has continuously been crafting new and improved designs for our x-ray tubes and decided it was the right time to upgrade our production line. We are looking forward to delivering high-quality systems and replacement tubes to customers for years to come.

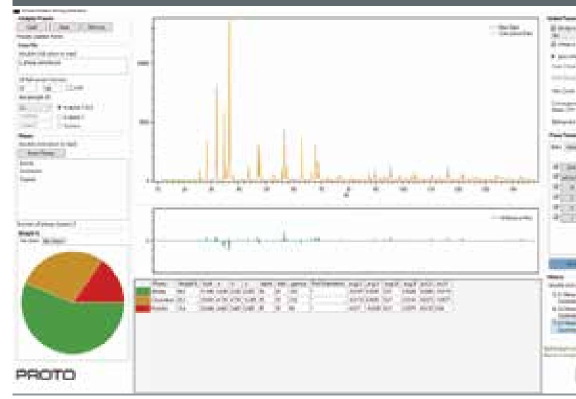
NEW & NOTABLE

recent innovations to the product line

LINE-FOCUS 32MM X-RAY TUBE

This new x-ray tube enables line-focus powder diffraction in tight spaces. Ideal for portable, custom, or compact systems.





RIETVELD SOFTWARE

Proto's new Rietveld package, PDAnalysis WPF, allows users to perform whole pattern fitting on acquired diffraction patterns and quantitatively identify phases in the sample.

ANTON PAAR
HTK 16N /HTK 2000N
STRIP HEATER CHAMBERS

NON-AMBIENT DIFFRACTION IN THE AXRD THETA-THETA

The AXRD Theta-Theta brings affordable non-ambient diffraction to your lab without occupying the space of a traditional laboratory diffractometer. Proto offers Anton Paar stages for easy integration into the AXRD Theta-Theta, allowing users to conduct powder XRD experiments at up to 2300°C.

TAKING THE PULSE OF MARINE ECOSYSTEMS

Climate change is widely viewed as one of the greatest challenges of our time. From clean electricity to paper straws, both governments and individuals have taken extensive measures to reduce its effects. The earth is currently experiencing a global warming event, which is resulting in a steady increase in global temperature, melting glacial ice, extreme weather, and substantial changes to our oceans, including rising sea levels and acidity. Over time, all of these effects can endanger both humans and wildlife, making climate change a topic that should be on everyone's radar.

Various methods have been used to quantify and track climate change, such as observing rainfall records, evaluating images taken from space, and taking measurements of seawater. By gathering data over a long period of time, scientists can make projections for the future and come up with measures to reduce the impact of climate change. Some of the commonly used methods have significant disadvantages, however; for instance, to measure the acidity of large portions of the ocean – not just of small water samples – scientists are at the mercy of computational models with their own variables and assumptions. The acidity of seawater is normally close to neutral but slightly basic (i.e., average pH of 8.2), but dissolved CO_2 from the atmosphere makes the water more acidic (lower pH). Even a small decrease in the pH of the ocean can have devastating effects on marine wildlife and cause species to dwindle or disappear. Thus, gathering accurate data from the whole ocean is crucial, but it is also a massive challenge – one that scientists are actively trying to solve.

> “BY GATHERING DATA OVER A LONG PERIOD OF TIME, SCIENTISTS CAN MAKE PROJECTIONS FOR THE FUTURE AND COME UP WITH MEASURES TO REDUCE THE IMPACT OF CLIMATE CHANGE.”

Dr. Aaron Celestian of the Natural History Museum of Los Angeles County (NHMLAC) is one of the scientists at the forefront of this research, along with his colleagues at the University of Southern California and Caltech. As the NHMLAC's curator of mineral sciences, Celestian specializes in mineral formation and interactions and how they might tell us more about climate change. “We can look at the stability of minerals in the oceans to learn more about the changing acidity of seawater through time,” says Celestian. To better understand the behavior of ocean minerals, Celestian has been studying ocean animals whose shells are formed from the carbon-bearing minerals aragonite and calcite, different crystalline forms (polymorphs) of

Photo courtesy of Jacki Moonves.

DID YOU KNOW?

Molluscs have the ability to control the morphology and orientation of biominerals that comprise their shells. For example, if calcite is available, some mollusc species use it for the outer shell layers, while they use aragonite for the soluble protein and ligament fiber portions of the shell.

FUN FACT

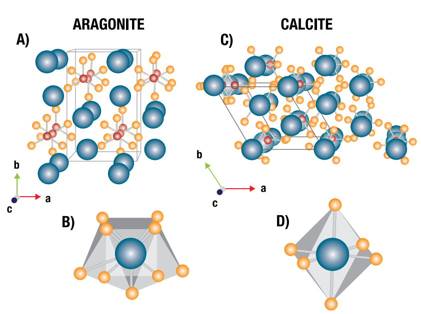
Among the atoms and molecules dissolved in the ocean are carbon (C), oxygen (O), sodium (Na), chlorine (Cl), magnesium (Mg), and calcium (Ca). Depending on the pH of the seawater, these components may be widely available or in short supply.

calcium carbonate. One of Celestian's primary interests is discovering just how quickly these minerals dissolve in seawater.

But how do the shells of ocean animals relate to ocean acidity and climate change? According to Celestian, the carbon-bearing minerals that are used to form shells (and corals) have varying degrees of susceptibility to acidic water. Aragonite, which has nine oxygen atoms bound to calcium, has weaker bonds than calcite; this means that it is more susceptible to low pH levels. Calcite, on the other hand, has six oxygen atoms bound to calcium, making its bonds stronger and less likely to break. However, when magnesium is present in the water, it often substitutes for some of the calcium atoms in calcite, distorting shell structures slightly and making them less stable. As Celestian notes, "In acidifying oceans, shells and other hard parts built from aragonite and Mg-calcite will be more readily dissolved and therefore more vulnerable."

This is an important relationship that Celestian and his research colleagues, the C DisK-IV (Carbon Dissolution Kinetics) team, have been investigating during a series of voyages across the Pacific Ocean. Their latest voyage took place during the summer of 2017 with the group traversing the Pacific from Hawaii to Alaska. While ocean pH and dissolved carbon have been measured in the past, this group's focus is identifying the concentration of carbonate minerals in seawater and measuring the rates at which these minerals dissolve. In their research, the team merges marine mineralogy with x-ray diffraction (XRD) to better characterize their samples, providing greater insight into ocean acidification over time.

As the ship moved along the path, the group used seawater pumps to filter water from several different depths and a multi-corer to collect sediments from the sea floor. The scientists then removed the filters and analyzed the contents using a Proto AXRD® powder diffractometer. Since powder XRD is ideal for identifying different crystalline



Structures of calcium carbonate polymorphs. Aragonite (left) has an orthorhombic crystal structure, while calcite (right) is trigonal.

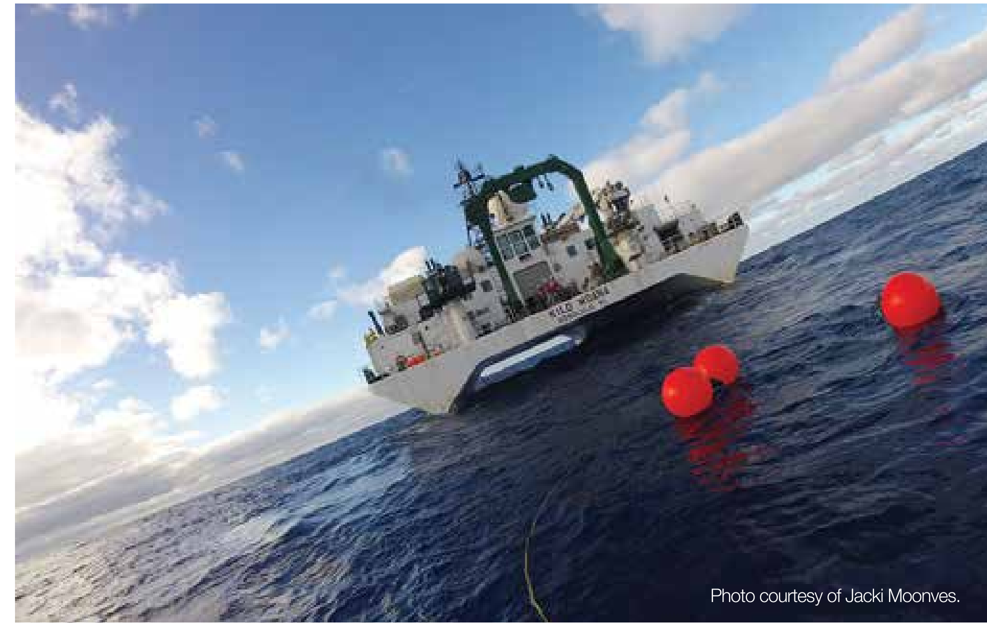


Photo courtesy of Jacki Moonves.

structures, the percentage of calcium carbonate polymorphs present could be identified at different ocean depths. Thus, the group was able to identify the ratio of aragonite to calcite in the water along their voyage path. In the water samples taken near Hawaii, they observed a greater depth at which aragonite is stable; however, as they moved toward Alaska, they observed aragonite stability at shallower depths. Because the aragonite was not stable below a depth of 200 meters, they concluded that these waters were more acidic.

As Celestian notes, "The more CO_2 in the atmosphere, the more carbonic acid is produced, and the lower the pH of the oceans." When the pH of the oceans gets lower, animals that form shells will have difficulty maintaining their structure, forcing them to expend more energy on shell formation and repair. As a result of these additional energetic demands, the animals may not be able to survive and reproduce, eventually causing a decline in their population. This is only one of the reasons why humans are making concerted efforts to reduce their carbon footprint – and XRD is poised to play an important role in the ongoing investigation of how mineral behavior informs ocean acidity.

Dr. Aaron Celestian of the Natural History Museum of Los Angeles County (NHMLAC)

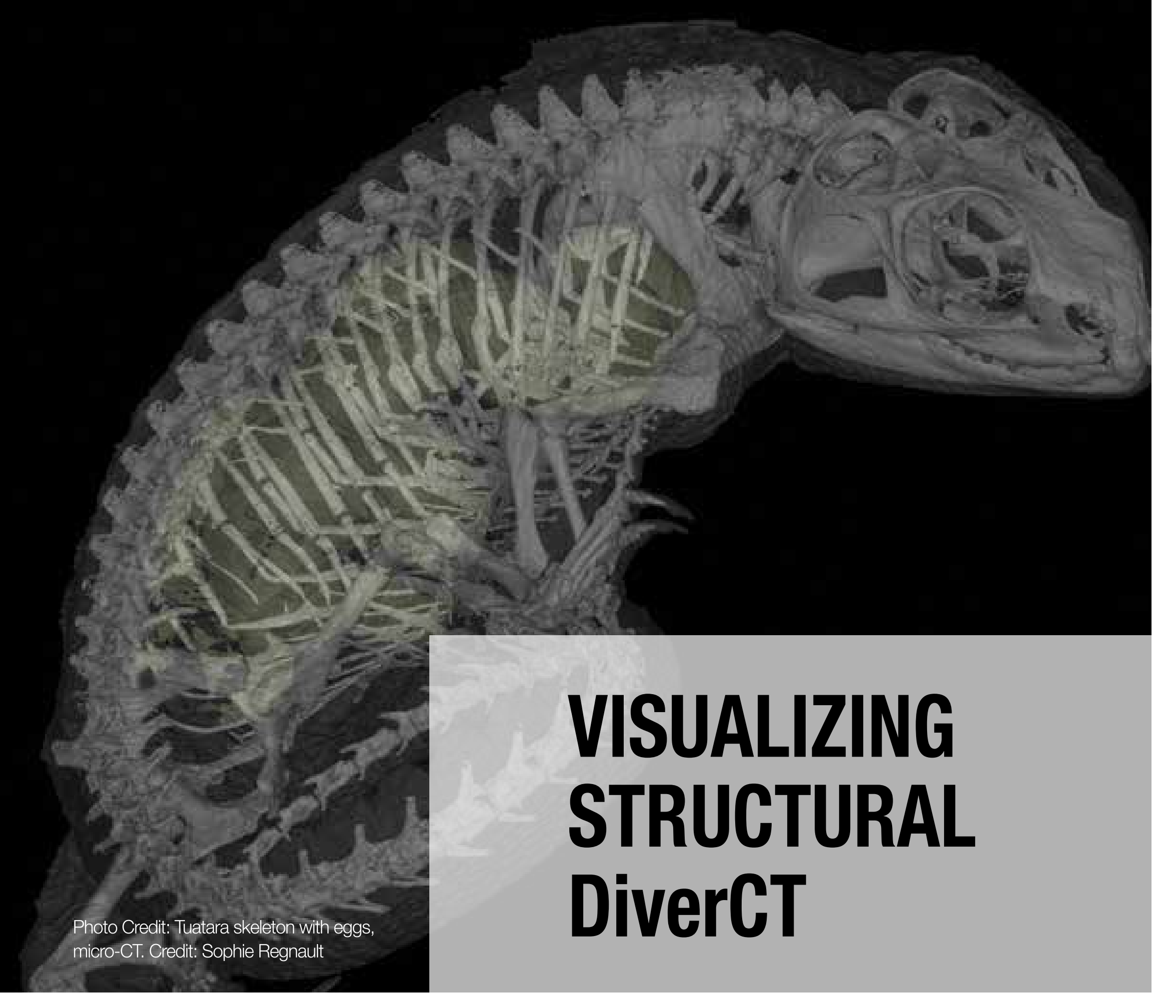


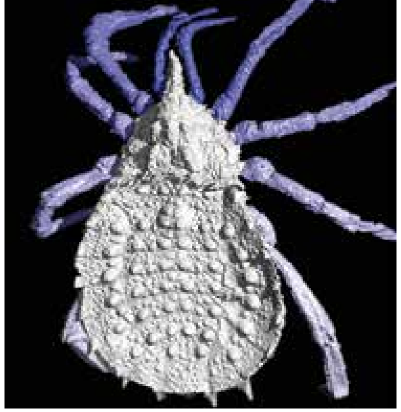
Photo Credit: Tuatara skeleton with eggs, micro-CT. Credit: Sophie Regnault

VISUALIZING STRUCTURAL DiverCT

For most people, computed tomography (CT) scanning is a familiar medical term for a diagnostic x-ray test. They may have even had a CT (or CAT) scan ordered by one of their doctors for investigation of the head, the abdomen, or another part of the body. However, the uses of CT scanning extend well past the medical field. These types of x-ray scans help us visualize the internal features of structures within many solid materials, including their shapes, sizes, and textures.

A single x-ray photograph is often taken to see into an object, with the resulting image being an average of the density of the material, a 3D object flattened into 2D. However, CT scanning involves a series of x-ray photographs being taken rotationally around an object, which can then be used to generate a 3D image. Conventional CT typically scans large areas, so the resolution is limited to about 1 millimeter due to limitations in the number of pixels on the x-ray detector; however, by scanning a smaller area, micro-CT scanners can provide much higher resolution – about 1000 times higher (1 micron). This level of detail is often needed to image small, complex structures, allowing researchers to make important discoveries in a variety of fields.

When fossilized segments of dinosaur tail were recently recovered in present-day Alberta, Canada, the scientists at Tel Aviv University noted cavities on the vertebrae that resembled a rare human cancer called Langerhans cell histiocytosis (LCH). By micro-CT scanning the tumor and surrounding blood vessels, they were able to confirm that the lesions present were a result of LCH. LCH results in distinctly shaped bone defects, and when the spine is involved, it typically affects the thoracic vertebral bodies (the oval bones between discs), which could cause anterior wedging and compression fractures. The researchers observed this pattern, identified LCH as the cause, and thereby concluded that LCH has existed for approximately 66 million years. The disease had never before been identified in a dinosaur or in a fossil. Thanks to studies like these, we are one step closer to determining the causes of certain illnesses, as well as the evolutionary conditions that enabled their development and survival.



Computed tomography (CT) scan reconstruction of a 312-million-year-old arachnid fossil. The original Plesiosiro madeleyi represents the only known specimen of the order Haptopoda, an extinct branch of the arachnids class. (Colourized)

Not only are micro-CT scans useful for identifying diseases, they can also reveal key information about the historic timeline of various animals. Researchers at the University of Missouri recently analyzed tubular fossils and discovered the world's oldest-known digestive tract. This 550-million-year-old fossil from the Nevada desert could be an important piece of the evolutionary puzzle, as the micro-CT imaging allowed for visualization of its small internal structures and revealed its anatomical similarity to worms; the organism, from the ***cloudinid family***, was previously thought to bear more anatomical

WHAT IS THE CLOUDINID FAMILY?

The cloudinids lived during the final 10 to 15 million years of the Ediacaran Period, which is the period that preceded the Cambrian Explosion. Since scientists believe that the Cambrian Explosion is the period in which an influx of complex animals suddenly appeared in the fossil record, discoveries from the Ediacaran Period have special phylogenetic significance (i.e., they can shed light on evolutionary relationships).

WHAT IS REFRACTION

When an x-ray wave interacts with matter, it is slowed down. Since the frequency of a wave always stays the same, the wavelength must decrease, and thus a phase shift (shift in wave peak positions) occurs relative to the original wave before it entered the medium.

similarity to corals. Because of technological advances in imaging, particularly micro-CT imaging, scientists can view 3D images of fossils and identify internal structures without destroying the sample.

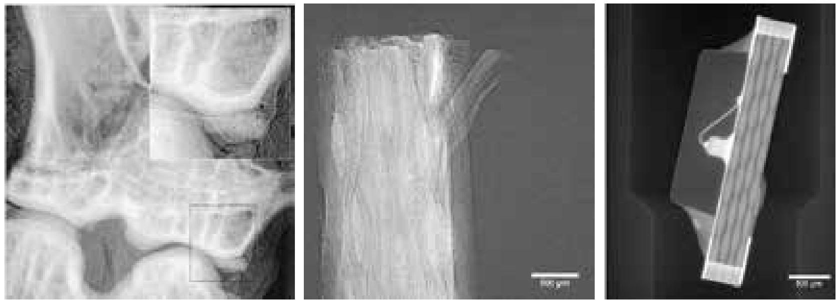
Given the important and diverse applications of micro-CT, there has been a big push for researchers to develop improved equipment with which to image samples. A company that is pushing the boundaries of micro-CT technology is KA Imaging, a spin-off from the University of Waterloo. Proto recently partnered with KA Imaging to develop their latest micro-CT scanner, the inCiTe, in which images are taken continuously while x-rays impinge upon a rotating sample. This system is the first commercial CT scanner to feature KA Imaging's unique, high-spatial-resolution amorphous-selenium (a-Se) detector. The compact benchtop design makes it possible to perform micro-CT scans without extensive space requirements. In addition, the a-Se detector has an 8-micron pixel size, which is much smaller than typical Si-based flat panels. This detector allows for direct conversion of x-ray photons into electrical charge. This direct conversion coupled with a high detective quantum efficiency (DQE) enables efficient imaging at low flux and high energy, making the inCiTe a very versatile machine.

Because of the detector's small pixel size, this system has the added advantage of phase-contrast imaging, which allows for the investigation of materials that cannot be visualized with conventional x-ray imaging. For example, soft biological tissues and polymers typically have poor absorption; however, when an x-ray passes through these materials, it is also ***refracted***. After leaving the object, the refracted (phase shifted) x-rays can start to interfere with each other, and when given a long enough propagation distance, the interference effects can be observed on an x-ray detector. The effect is most pronounced when there is an abrupt change in refractive index – for instance, at boundaries and edges within a material. Phase shifts' effects are typically 100 to 1000 times stronger than effects from absorption, thereby improving the

visibility of samples that typically have weak x-ray absorption. However, the interference effects occur over very small distances, so a high-resolution detector is needed in order to observe this edge enhancement.

For example, using the inCiTe system, tissues can be visualized with high contrast, such as in the mouse knee shown below (left), enabling the cartilage to be examined in great detail. The composite material pictured below (center) is a piece of Kevlar. With the help of phase-contrast imaging, individual fibers sized 10-20 μm can be seen within the material. The third CT image below is an LED. Phase-contrast capabilities allowed its substrate layers to be identified, and the wire bond shapes can be seen clearly thanks to the system's high-resolution detector.

The inCiTe system is ideal for analyzing fine structures in materials such as polymer composites and biomaterials but can be used in various fields and applications, including non-destructive testing (NDT), electronics, additive manufacturing, geology, and agriculture.



THE STRESSES OF AIRCRAFT SAFETY

When an aircraft component is manufactured, great care is taken to ensure the accuracy of the material's size, shape, and properties. Given the importance of these parts in maintaining the safety of aircraft, the consequences would be dire if the proper safety measures were not strictly adhered to. However, quality control processes do not just apply to the outward characteristics of the component; during the machining and fabrication of critical parts, invisible changes can occur within the material and render it unsafe after a period of use – namely, these would be changes to the residual stress state of the part. Because of the very likely possibility of introducing residual stress into an aircraft component during various stages of manufacturing, it is highly important for manufacturers and suppliers to understand what residual stress is, how to detect it, and how to ensure the safety of parts before and during use.

Residual stress is the internal stress locked into a component after all external loads have been removed. These stresses occur when a material establishes equilibrium after undergoing plastic deformation due to either inelastic mechanical deformation, thermal loads, or phase changes. Residual stress can be either beneficial or harmful to a component. Compressive stresses are generally favorable, as they push the material together, while tensile stresses tend to be unfavorable, as they pull the material apart. In the absence of an external load, the total sum of all residual stresses in a part is always zero. More specifically, the net residual stress across any cross section is always zero; however, the distribution of stress can vary greatly. Therefore, identifying areas of high residual stress is critical in preventing premature failures.

It is also important to remember that residual stress is not the only stress that will be present in an aircraft component; during use, the part will also be subjected to applied stress, and the total stress present in a material is equal to the residual stress plus the applied stress. This is particularly important when the residual stress distribution results in high tensile stress in a region that will be subjected to high tensile loading, and similarly in regions of high compressive residual stress subjected to compressive loading.

When stress levels in aircraft components are too high, several different failure modes can pose problems. Firstly, ***stress corrosion cracking*** (SCC; also known as environmentally assisted cracking) is a potential source of failure that involves sustained tensile stress above a material's SCC threshold. Another common issue is fatigue, which can be divided into low-cycle and high-cycle fatigue (LCF and HCF). The terms are so named because of the number of cycles it typically takes for failure to occur – about 10^4 to 10^5 cycles for LCF and upwards for HCF. LCF typically involves high levels of localized loading, resulting in cumulative damage to a part and multiple crack initiation sites, along with changes in residual stress throughout the component's service life. On the other hand, HCF tends to involve lower levels of applied loading, eventually leading to a single crack initiation site caused by a defect, resulting in more abrupt changes in the residual stress state toward the end of the component's life. Other factors to consider are erosion – especially the combined effects of erosion and fatigue cracks – and creep, the tendency of a solid material to move slowly or deform permanently due to persistent mechanical stresses. Over time, creep can result in fractures via creep rupture, and this process can be accelerated by high tensile stresses.

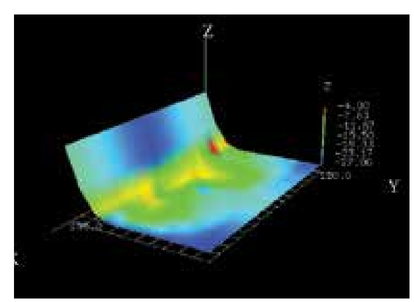
Because so many failure modes are impacted by detrimental residual stress, manufacturers and suppliers need a reliable method of investigating residual stress states in as-manufactured components and predefined processes for repairing or replacing in-service components before failure occurs. Since high levels of tensile stress can contribute significantly to SCC, fatigue failure, and creep crack initiation, introducing beneficial compressive stress is a possible solution. The method chosen depends on the specific application, but some examples include shot peening, laser shock peening, burnishing, ultrasonic impact treatment, overspinning, and split-sleeve cold expansion. Some of these processes take place during fabrication of the components, while others have the potential to introduce favorable stress during service life – for example, during tear-down and inspection. When sufficient compressive stress is introduced in failure-critical regions, the in-service applied stress can be overcome, and the total magnitude of residual stress can be reduced to below the SCC threshold, fatigue limit, or creep rupture strength.

In order to determine the residual stress state of a component and prevent premature failure, a reliable, quantitative measurement method must be chosen. A widely accepted quantitative method that can measure residual stress without damaging the component is x-ray diffraction (XRD). XRD uses the distance between crystallographic planes (d-spacing) as a strain gauge. When a material is in tension, the d-spacing increases, and when a material is in compression, it decreases. The diffraction angle (2θ) is measured experimentally, and the d-spacing is calculated using Bragg's law: $n\lambda=2d\sin\theta$, where λ is the source wavelength and n is an integer multiple of the wavelength. XRD measures at and near the surface, which is necessary for investigating fatigue and SCC-related problems, since the near-surface stresses are most important in such applications. In addition, XRD can be portable, making it an efficient choice for repeated and/or field measurements.

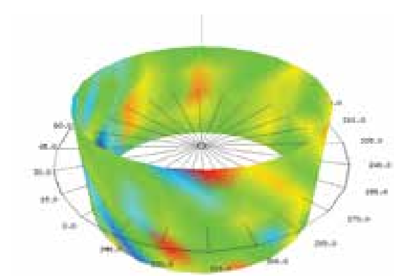
In a real-world application of residual stress testing using XRD, measurements were taken on airplane frames that were susceptible to stress corrosion cracking. According to the given specifications, the frames had been shot peened prior to testing. However, measurements performed both in the lab and in the field revealed that surface residual stress gradients were present, along with locally varying magnitudes of residual stress. These results were not consistent with shot peened components, as tensile stresses were identified instead of compressive stresses; in addition, shot peened components tend to exhibit homogeneous (compressive) surface residual stress rather than gradients. Based on these results, it could be determined that residual stress had not been adequately controlled upon fabrication of these components.

SCC

Along with high tensile stress, a susceptible material and a corrosive environment can contribute to SCC; however, these two factors can be difficult to alter. For example, removing the component from its environment is usually impossible, and adding a coating to effectively remove it from its environment is likely to be a short-term solution due to degradation of the coating.



Residual stress map on an aircraft frame



Residual stress map inside a trunion

TRAVEL TIPS

FOR BUSINESS TRIPS

#1 GET A PORTABLE POWER CHARGER

#2 INSTALL AND USE YOUR LOYALTY APPS

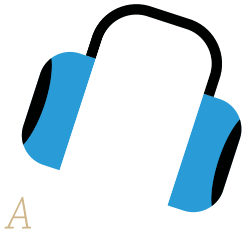
#3 TAKE PICTURES OF YOUR RECEIPTS

#4

BRING SMALL BILLS FOR TIPS

#5 PICK ONE THING YOU WANT TO SEE AND REALLY ENJOY IT

#6



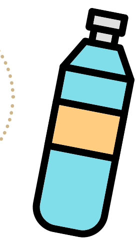
INVEST IN A PAIR OF NOISE-CANCELING HEADPHONES

#7 STRETCH BETWEEN FLIGHTS

#8 PLACE A DRYER SHEET IN YOUR SUITCASE TO REDUCE STATIC

#9 RESEARCH BUSINESS ETIQUETTE OF YOUR TRAVEL DESTINATION

#10



STAY HYDRATED DURING FLIGHTS

Photo credit: Dan Boshart.

RIGHT BRAIN UNLEASHED

the artistic side of Proto

Matt Williams brings a unique set of skills to the Proto team. As Physicist and Software Developer, he programs most of the day, but he turns the music on at night. Matt plays bass with a group of impressively skilled musicians, all working toward the singular goal of bringing back the funk. He is also a classically trained cellist and performs with the Windsor Symphony Community Orchestra.

#FUNSCIENCE

when you need a break from the lab

A YEAR OF SCIENCE

celebrations & discoveries of 2019/2020

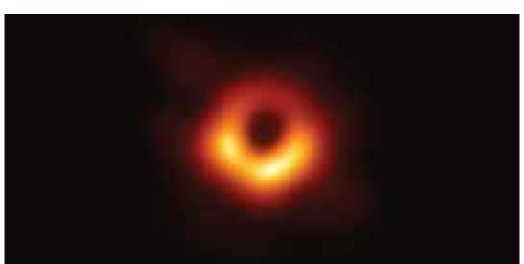
HAPPY 125TH ANNIVERSARY

When Wilhelm Röntgen discovered x-rays in 1895, they were put to use in the medical field almost immediately. However, at the time, little was known about these electromagnetic energy waves. Now, 125 years later, x-rays have enabled advancements in many fields, including diffraction.

BLACK HOLE

Scientists recently captured the first ever image of a black hole and its shadow. The image shows the supermassive black hole in the center of the Messier 87 (M87) galaxy, which is about 55 million light-years from Earth.

Photo courtesy of Event Horizon Telescope Collaboration.



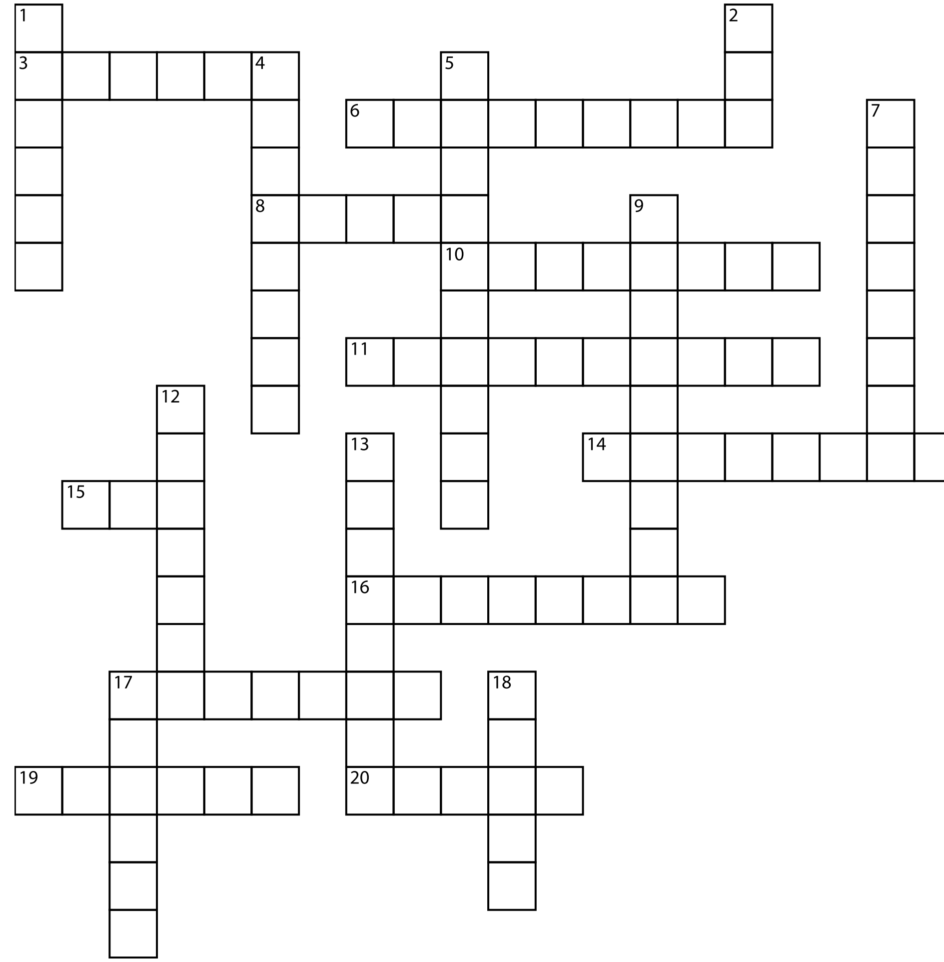
GIANT SQUID SIGHTING

In June 2019, the nearly lost species was spotted in its deep-sea habitat in the Gulf of Mexico. Scientists were able to capture the first video of a giant squid in U.S. waters thanks to their special red-light underwater camera.

A giant squid, at least 3 to 3.7 meters (10 to 12 feet) in length, approaches the Medusa's e-jelly lure before realizing the e-jelly is not food and retreating.

Photo courtesy of Edie Widder and Nathan Robinson – Ocean Research & Conservation Association (ORCA).

CROSSWORD



ACROSS

3. Red litmus
6. No worm guts, no glory
8. Second-largest violin family member
10. Inclination
11. Ear speakers
14. Dental damage
15. Vehicle
16. Pointing out differences
17. Mass per volume
19. Hollow receptacle
20. Smallest image element

DOWN

1. Elite combatant
2. Rotten fruit
4. Knuckle habit
5. Radiological sectioning technique
7. Shocking element
9. Present in the Park but not the Ark
12. Uppermost layer
13. Black resin work area
17. Cactus land
18. Radiohead weirdo

Hint: *For help with the more challenging clues, read the magazine pages carefully!*

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