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hen Arvid Pasto  
first heard about

an amazing surface treatment in 2001 from a lawyer named Mark Deininger, his first thought was, "This sounds like snake oil." But Pasto, then director of the High Temperature Materials Laboratory at Oak Ridge National Laboratory, changed his mind after talking to a long-time trustworthy friend at the same scientific conference in Pittsburgh where Deininger personally described the innovation. The friend is a consultant to Deininger's company.

Soon thereafter, Pasto arranged for Deininger, president and chief executive officer of C3 International, to come to Oak Ridge to meet with group leaders of materials sciences and technologies.

From 2002 to 2006, the unique surface treatment underwent a variety of tests involving a half dozen researchers. ORNL researchers were amazed that a Russian inventor had found a way to attach rare-earth elements to an organic chain. When the organic compounds leave, the rare earth left behind bonds to the substrate as nanocrystals pin down the element.

The ORNL researchers validated the properties of this remarkable surface treatment that Deininger describes as "an implantation that anchors a nanofilm." Blue and others call it a "molecular infusion, or implantation, surface treatment," or MIST.

The surface treatment contains 3-nanometer crystallites that plug the thin oxide film into the grain boundaries of a bulk material's surface, making the material extremely resistant to wear so it lasts longer. ORNL researchers measured the dimensions of the crystallites that make

High-power plasma  
arc-based lamp

**Novel surface treatments are greatly  
increasing the durability of industrial tools.**

# Miraculous

the ultrathin film adhere extremely tightly to the surface. No other "coating" has particles this small that bind to a surface.

Deininger lists other extremes associated with the special surface treatment. "MIST is extremely easy to apply by dipping or spraying and extremely cost effective and economical," he says. "The coating is extremely green and environmentally friendly. It is also extremely versatile because any one of 97 different elements can be used in MIST on a variety of surfaces, from metals to ceramics to glasses to carbides. The permutations add up to more than 200 million properties."

Today the American company that grew out of a Russian discovery has a research facility on the ORNL campus—the first private company to be housed at a

national laboratory. Opened for business under Pasto's leadership on March 19, 2008, C3 International is a tenant of the Oak Ridge Science & Technology Park.

When Deininger came to ORNL in 2002, he gave a presentation on his company's solution-based technique for depositing ultrathin films of rare-earth oxides and other elements. Researchers were attracted by the processing temperature that anchors the nanoscale coating to an underlying substrate—a temperature on the order of 400°C that could be easily achieved by infrared light from a tungsten-halogen lamp in the lab of ORNL's Craig Blue, then leader of the Laboratory's materials processing group.

Pete Angelini, then manager of the Industrial Technologies Program, said





# Coatings

to Blue, "Let's see if Mark can pass the aluminum die-casting test. No one has yet proven that a coating can handle the liquid metal attack."

A user agreement was set up, allowing Deininger to work with researchers at the High Temperature Materials Laboratory and in Blue's high bay to evaluate the ultrathin coating. Blue applied Deininger's coatings to steel thermocouple sheaths that were then immersed in aluminum.

To make an aluminum automobile part, for example, hot molten aluminum must be poured into a steel mold to obtain a specific shape. Casting aluminum components in steel dies is a problem because hot aluminum has an affinity for sticking, or soldering, to the steel in these molds. "You have to

sandblast the aluminum off the steel die after you open it so the mold can be used again," Pasto says.

Blue dipped a steel thermocouple sheath in Deininger's cerium oxide liquid and then plunged the coated sheath in molten aluminum. He compared the sheath's resistance to attack from the aluminum to that of an uncoated sheath by measuring how much aluminum adhered to the surface. When he dipped the sheath in the coating liquid twice, he found that the sheath lasted twice as long as the uncoated sheath; in other words, the durability increased 100%. When he dipped the coated sheath three times, 200%; 4 times, 300%; eight times, a 700% increase in life extension. Stated differently, Blue observed an almost

direct linear correlation between the thickness of the ultrathin coating and the durability of the coated component.

Researchers at HTML used Auger spectroscopy, X-ray diffraction and transmission electron microscopy to evaluate the surface treatment and its ability to increase the wear resistance of coated objects. Studying a zirconium oxide coating, they obtained beautiful TEM images showing that the sizes of the zirconia particles ranged from 3 to 5 nanometers.

Deininger then tried zirconium oxide coatings for the aluminum-and-steel challenge. He used a steel pin normally used to push a molded aluminum part out of the steel die. The molten aluminum tends to stick to both the die and the steel pushout pin.





*C3 International President Mark Deininger (right) with Arvid Pasto*

"The easiest way to test whether the C3 zirconium oxide film effectively resists aluminum attack on steel is to characterize the coated pushout pin," Pasto explains. "Mark proved that the coated pin resisted aluminum attack and could be used over many times without need to blast the aluminum off the steel pin." C3 International passed the test.

"We made great strides in aluminum die casting and filed patents on this application in 2003," Deininger says. But pressures on the U.S. automotive industry made it difficult for C3 to penetrate a highly competitive market.

"We have a business model at C3 International to look for partners that are strategically located in an industry to scale up our surface treatment technology," Deininger explains. "In late 2007 we signed a license agreement with Magna-Tech Manufacturing in Indiana, which treats aluminum parts coming out of the steel dies and is involved with every major aluminum diecaster in North America. The company does not make the dies.

"We are training Magna-Tech employees how to apply the custom-engineered chemical surface treatment. The coating is designed to extend the life of the die tools from which aluminum components are made as well as the parts themselves."

## Under the Bright Lights

Few things attract our attention like a sudden burst of light at night. In Oak Ridge, researchers have the capability not only to produce but also control intense lighting in the lab coming from the world's most powerful plasma arc-based lamp.

For eight years Craig Blue has been demonstrating the potential industrial applications of rapid radiant heating using flashes of infrared light from tungsten-halogen lamps and then shorter flashes of light from a high-power plasma arc-based lamp (see photograph on pp. 4-5). Vortek Industries of Canada manufactured the early arc-based lamps. Mattson Technologies, a California company, purchased Vortek in 2004.

Blue manages industrial technologies and materials processing at Oak Ridge National Laboratory, which owns two

high-power plasma arc-based lamps. Blue has advised Mattson on how the lamp can be used as a research tool.

By adjusting lamp-processing parameters such as flash time and power densities, Ron Ott can reach several extremes in a pursuit to achieve advances in flexible electronics.

"We can flash the lamp down to 1 millisecond while discharging 12 megawatts, providing to the surface 20,000 watts per square centimeter and heating the surface on the order of 1 million degrees per second. Only nature can exceed these extremes."

Ott leads a group that explores the potential of the high-power lamp's photons in helping to fabricate more-efficient, lower-cost thin-film batteries, thin-film transistors and photovoltaic cells for converting the energy of sunlight into electricity.



Chaitanya Narula, leader of ORNL's Physical Chemistry of Materials group and a catalyst chemist, has found ways to use engineered C3 surface treatments on particulate exhaust filters for diesel engines. As a result, the traps use less energy to remove carbon and last longer. A similar application of the coating in oil refinery cokers slows carbon buildups, resulting in reduced releases of carbon dioxide to the atmosphere.

petrochemicals, fuel cells, microelectronics, solar energy and food processing.

## Boring in

Although Georgians experienced a severe drought in 2007, they are not far removed from periods of heavy rain and flooding. One problem plaguing Atlanta had been the inability to store and treat combined sewage and storm-water

back out and the disc cutters are replaced—an expensive procedure. During the experiment the disc cutters at the front end had been coated with a material developed by ORNL materials researchers. As hoped, the tunnel boring machine penetrated deeper than usual through the mountain rock.

ORNL's Bill Peter and Craig Blue have developed an iron-based, 200-micrometer-thick, nanocrystalline coating formed by heating amorphous powders with laser light. The coating's specific application is designed to increase the lifetime of the disc cutter rings in tunnel boring machines. Preliminary results indicate that the novel coating extends the lifetime of the disc cutter 20 to 30%.

The researchers put the amorphous powder into a polymer binder and sprayed the powder onto a disc cutter. Blue explains that the extremely high heating and cooling rates of their laser-based technique change the non-crystalline bulk metallic glass powder into a nanocrystalline structure, while creating a metallurgical bond between the coating and the steel cutter substrate. The strength of the bond prevents the coating from spalling, or breaking into chips.

"The laser, in effect, heats the glassy coating and a small layer of the substrate beyond the melting temperature," Peter says. "Convective stirring occurs, changing the chemistry of the coating. An extremely high cooling rate prevents grains from coarsening, producing a

*Continued on pg. 8*

**"The ORNL coating achieved a 20 to 30% increase in disc cutter lifetime despite the presence of mountain rock such as gneiss and granite."**



"The range and scope are extremely small and extremely large at the same time," says Pasto. "We can spray this surface treatment with nanofilm dimensions on extremely large industrial tools in steel and petrochemical plants. Applying a nanofilm onsite to stainless steel tubes and huge rollers is unheard of."

Deininger asserts that MIST is extremely divergent in its applications, based on the number of industries that will likely find uses for the technology: steel and aluminum for the automotive industry, particulate exhaust filters for diesel engines,

overflows from major rainfall events. Two tunnels have been built to capture and store the overflows and then convey the polluted water to treatment facilities, preventing sewage from entering and contaminating area rivers.

In 2007 when the tunnels were being completed, an experiment was under way. Normally, a Herrenknecht tunnel boring machine chews through mountain rocks until its steel disc cutter rings are smashed into metallic hexagons too worn to crush rock. When the cutters are no longer performing well, the machine is pulled

"Our focus is to do high-temperature processing of non-crystalline silicon over broad areas on low-temperature substrates," Ott says. "Non-crystalline silicon and silicon-germanium layers are cheaper than crystalline silicon layers.

"We have shown that we can initiate solid-phase crystallization, which will introduce a nanocrystalline structure with fewer defects and higher efficiencies. The goal is to optimize the microstructures to improve photon collection efficiencies for solar cells while not altering the underlying substrate."

Furthermore, the lamp's flash of light will heat only the surface layers to extremely high temperatures. The substrate will barely be heated at all. That means the underlying layer, which is usually metal to withstand high-temperature processing, can be replaced with a

cheaper plastic substrate capable of bending and conforming to a desired shape, giving rise to the term "flexible electronics."

In 2004 Blue, Queen City Forging in Cincinnati, Ohio, and others received an R&D 100 award for an optimized combination of radiant and convection heating for processing materials. The forging company now uses this technology to make lightweight aluminum forged components to replace more expensive and heavier titanium and other metal parts for aerospace and automotive applications.

In February 2008, producers and cameramen with the cable television program "Modern Marvels" came to ORNL to film the high-power plasma arc-based lamp for a feature on ultrahigh-temperature heating that will likely grab viewers' attention.—C.K.



*Continued from pg. 7*

nanocrystalline composite coating with high wear resistance.

"We heat and cool the coating at thousands of degrees per second. We found that laser fusing these coatings works well on many steel substrate components that see high wear rates, such as drill bits and disc cutters."

The chemical composition of the coating came from a program they worked in that was jointly funded by the Defense Advanced Research Projects Agency and the Department of Energy's Office of Civilian Radioactive Waste Management/Science and Technology program. The goal was to develop coatings with high corrosion resistance for the steel canisters that will confine high-level radioactive waste at Yucca Mountain in Nevada.

"This is the first time that a coating applied to a disc cutter survived the extreme environment of boring a tunnel," Peter says. "Our coating was first tested at a one-of-a-kind linear cutting machine at the Colorado School of Mines. The machine simulates loads in a tunnel. We used a video camera and still-shot camera to monitor the changes in disc cutter coatings.

However, we obtained 'real' laboratory results when we took cross sections of tested disc cutters and analyzed their coatings."

The experiment was also the first time that any coating survived in this machine, and the School of Mines has been testing coatings for 30 years. The benchmark was one the ORNL researchers had to pass before they could test their coated cutters in the field.

Operating a \$25 million tunnel boring machine costs \$100,000 to \$150,000 a day. Peter rode on the machine testing the ORNL coating on four disc cutters.

"This machine is monstrous," he says. "Tunnel boring machines are like small factories traveling underneath the ground. Machines can be as large as 50 feet in diameter and over 100 feet long with more than 50 circular cutters. Each disc cutter measures 17 inches in diameter and weighs 60 pounds.

"The boring machine pulverizes rock directly underneath the disc cutter, causing the rock to crack. After two cracks intersect, a chunk of rock is removed. Hydraulic rams push the machine forward against the rock wall at five-foot inter-

vals. The machine lays down electrical wires and track as it puts up the tunnel. The humidity was so high in this extreme environment that I could not take photos of the disc cutters up front."

About 45% of a tunnel boring machine's downtime is due to changing out disc cutters as a result of wear. Herrenknecht uses a proprietary H-13 steel for their \$350 disc cutters. The company is interested in finding a coating that costs no more than 10% of the disc cutter price and increases lifetime by 20 to 30%.

"At the Atlanta job site we showed that four disc cutters with the ORNL coating achieved a 20 to 30% increase in lifetime despite the presence of mountain rock such as gneiss and granite," Peter says. "Some of our coating remained on the disc cutters after they traveled 300,000 linear feet through rock. A competitive coating failed immediately, but our coating was still on the disc cutter after seven five-foot pushes.

"The Atlanta site is considered significant because it has some of the worst conditions that a boring machine could face. The disc cutters with our coating showed slow abrasive wear. Many of the disc cutters without a coating lost significant chunks of metal."

The ORNL team will test coated disc cutters under more typical mountain conditions, such as abrasive slurry, which has the consistency of toothpaste. An even greater improvement in the lifetime of coated disc cutters is expected under these conditions.

"Martin Herrenknecht, founder of Herrenknecht about 30 years ago, visited ORNL recently to look at our coating technology and was impressed by the money-saving potential," Blue says. "Through the Work for Others program here, he funded further testing of our coating and bought 20 disc cutter rings for us to coat and test."

Peter will coat 20 rings and test them on Herrenknecht tunnel boring machines as they bore the Gotthard Tunnel underneath the Swiss Alps.

The 11-mile-long Gotthard tunnel will be the longest running tunnel in the world. Preliminary tests suggest that, in the extreme environment of the Swiss Alps, the ORNL coating is likely to pass another benchmark test and is even less likely to be passed off as snake oil.—Carolyn Krause

**"We heat and cool the coating at thousands of degrees per second, which works well on many steel substrate components that see high wear rates, such as drill bits and disc cutters."**

