Materials & Coatings

Ambient Dried Aerogels

NASA's Jet Propulsion Laboratory, Pasadena, California

A method has been developed for creating aerogel using normal pressure and ambient temperatures. All spacecraft, satellites, and landers require the use of thermal insulation due to the extreme environments encountered in space and on extraterrestrial bodies. Ambient dried aerogels introduce the possibility of using aerogel as thermal insulation in a wide variety of instances where supercritically dried aerogels cannot be used. More specifically, thermoelectric devices can use ambient dried aerogel, where the advantages are in situ production using the cast-in ability of an aerogel.

Previously, aerogels required supercritical conditions (high temperature and high pressure) to be dried. Ambient dried aerogels can be dried at room temperature and pressure. This allows many materials, such as plastics and certain metal alloys that cannot survive supercritical conditions, to be directly immersed in liquid aerogel precursor and then encapsulated in the final, dried aerogel. Additionally, the metalized Mylar films that could not survive the previous methods of making aerogels can survive the ambient drying technique, thus making multilayer insulation (MLI) materials possible. This results in lighter insulation material as well.

Because this innovation does not require high-temperature or high-pressure drying, ambient dried aerogels are much less expensive to produce. The equipment needed to conduct supercritical drying costs many tens of thousands of dollars, and has associated running expenses for power, pressurized gasses, and maintenance. The ambient drying process also expands the size of the pieces of aerogel that can be made because a high-temperature, high-pressure system typically has internal dimensions of up to 30 cm in diameter and 60 cm in height. In the case of this innovation, the only limitation on the size of the aerogels produced would be in the ability of the solvent in the wet gel to escape from the gel network.

This work was done by Steven M. Jones and Jong-Ah Paik of Caltech for NASA's Jet Propulsion Laboratory. For more information, contact iaoffice@jpl.nasa.gov. NPO-49008

Applications for Gradient Metal Alloys Fabricated Using Additive Manufacturing

A new roadmap for gradient metals that could be used in cars, optics, aircraft, and sporting goods.

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Recently, additive manufacturing (AM) techniques have been developed that may shift the paradigm of traditional metal production by allowing complex net-shaped hardware to be built up layer-by-layer, rather than being machined from a billet. The AM process is ubiquitous with polymers due to their low melting temperatures, fast curing, and controllable viscosity, and 3D printers are widely available as commercial or consumer products. 3D printing with metals is inherently more complicated than with polymers due to their higher melting temperatures and reactivity with air, particularly when heated or molten. The process generally requires a high-power laser or other focused heat source, like an electron beam, for precise melting and deposition. Several promising metal AM techniques have been developed, including laser deposition (also called laser engineered net shaping or LENS® and laser deposition technology (LDT)), direct metal laser sintering (DMLS), and electron beam free-form (EBF). These machines typically use powders or wire feedstock that are melted and deposited using a laser or electron beam. Complex net-shape parts have been widely demonstrated using these (and other) AM techniques and the process appears to be a promising alternative to machining in some cases.

Rather than simply competing with traditional machining for cost and time savings, the true advantage of AM involves the fabrication of hardware that cannot be produced using other techniques. This could include parts with "blind" features (like foams or trusses), parts that are difficult to machine conventionally, or parts made from materials that do not exist in bulk forms. In this work, the inventors identify that several AM techniques can be used to develop metal parts that change composition from one location in the part to another, allowing for complete control over the mechanical or physical properties. This changes the paradigm for conventional metal fabrication, which relies on an assortment of "post-processing" methods to locally alter properties (such as coating, heat treating, work hardening, shot peening, etching, anodizing, among others). Building the final part in an additive process allows for the development of an entirely new class of metals, so-called "functionally graded metals" or "gradient alloys." By carefully blending feedstock materials with different properties in an AM process, hardware can be developed with properties that cannot be obtained using other techniques but with the added benefit of the net-shaped fabrication that AM allows.

Functionally graded metal alloys have been demonstrated previously using the LENS[®] process but the technique has not been used to develop functional hardware because the fabrication of robust gradient compositions is not trivial. In most cases, one cannot simply add a feedstock metal to another and expect to develop a new metal alloy free of cracks and unwanted phases. Developing gradient metal alloys requires a significant amount of knowledge in phase transformations to avoid compositions where brittle intermetallic compounds form (which may crack and, thus, destroy the hardware). To address this, the inventors have developed a technique where potential gradient compositions can be simplified and transposed onto a three-component (ternary) phase diagram (or a 3-dimensional hybrid phase diagram, in some cases) and the desired gradient composition can be "mapped"

to avoid unwanted phases (see figure). This process is part of a broader roadmap for gradient alloys that the inventors have developed to go from a desired combination of properties to a final part. This process proceeds by (1) identifying a combination of desired properties and modeling a component, (2) selecting metal alloys that satisfy the desired mechanical or physical properties, (3) developing a composition map to transition from one alloy to the other without forming unwanted phases, (4) selecting the desired AM building process, and (5) fabricating the part. Although at the time of this reporting it has not yet been demonstrated in the literature, the authors have identified a number of AM techniques that can be used to fabricate gradient metals, including laser deposition, laser sintering, wire free-form, thermal spray coating, metal dipping, among others. In any additive process involving metal feedstock, gradient alloys are possible.

In the current program, funded by NASA's Jet Propulsion Laboratory, the inventors use the gradient alloy roadmap to explore several desirable spacecraft applications. The LENS® technique was selected to develop gradient compositions free of brittle phases and those alloys were fabricated into prototype hardware. In one application, a gradient alloy mirror mount was developed that transitions between stainless steel at the base and Invar (an FeNi alloy) at the top. In space optics applications, glass mirrors are often bonded with epoxy to metal mirror mounts,



Various Aspects of the Innovation: (a) An isothermal slice of the Fe-Ni-Cr ternary phase diagram showing how different gradient compositions can be mapped. The lines represent composition gradients between 304L stainless steel and Invar 36, a simplified Inconel 625 alloy and a NiFeCr alloy. In some gradients, the path intersects brittle intermetallic phases, which can be avoided by changing the path to go through more desirable phases (the segmented green line). (b) An isogrid mirror fabricated using a 3D plastic printer and (c) the same part fabricated using LENS after some finish machining. The mirror surface is made of Invar 36 and the isogrid backing is a gradient alloy that transitions from Invar 36 to stainless steel. (d) A gradient alloy mirror assembly with a metal-coated glass mirror attached to the Invar side of the assembly using epoxy. The mirror assembly transitions into stainless steel at the base. (e) Test samples of a Ti-V gradient alloy being fabricated by LENS. (f) The compositions (as measured through electron dispersive XRD) of the gradient mirror assembly in (d) showing the transition between Invar and stainless steel. (g) A plot of hardness and thermal expansion across the gradient mirror assembly. The intermediate phases of the gradient have been designed to be soft austenite (as demonstrated by the decreased hardness). The controllable thermal expansion makes this part alluring for optics applications. One side of the gradient has a near-zero thermal expansion while the other side matches steel.

which are then bonded or fastened to an optical bench. When the part is exposed to extreme cold, the epoxy holding the mirror can crack and the fasteners holding the mirror mount can shrink, shifting the position of the mirror. In the gradient alloy, the glass mirror can be bonded to Invar, which has a near-zero thermal expansion coefficient that matches glass. However, the whole part does not need to be made from Invar, but rather it can be graded to stainless steel and then welded to the optical bench, eliminating thermal expansion mismatch from dissimilar metals. The gradient technique has also been used in an optics application to fabricate an Invar mirror with a high-stiffness isogrid backing. Isogrids are extremely costly and complicated to fabricate, but the AM technique allows gradient compositions to be built up right on the backside of the mirror. Other gradients have been developed, including a stainless steel to Inconel (a high-temperature Ni alloy) gradient to be fabricated into a valve stem for automotive applications. Invarcontaining metal inserts have been developed to eliminate low-temperature pull-out in carbon fiber panels and lowdensity titanium alloys have been graded to refractory metals (e.g. Nb and V) for high-temperature applications (such as rocket nozzles and engine components). Ongoing work has focused on developing new types of gradient armor for defense applications as well as a wide assortment of commercial applications.

This work was done by Douglas C. Hofmann, John Paul C. Borgonia, Robert P. Dillon, Eric J. Suh, Jerry L. Mulder, and Paul B. Gardner of Caltech for NASA's Jet Propulsion Laboratory. For more information, contact iaoffice@jpl.nasa.gov.

In accordance with Public Law 96-517, the contractor has elected to retain title to this invention. Inquiries concerning rights for its commercial use should be addressed to:

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Passivation of Flexible YBCO Superconducting Current Lead With Amorphous SiO₂ Layer

The aim of this project is to design and construct leads from YBCO composite conductors to reduce the heat load to adiabatic demagnetization refrigerators.

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Adiabatic demagnetization refrigerators (ADR) are operated in space to cool detectors of cosmic radiation to a few 10s of mK. A key element of the ADR is a superconducting magnet operating at about 0.3 K that is continually energized and de-energized in synchronism with a thermal switch, such that a piece of paramagnetic salt is alternately warm in a high magnetic field and cold in zero magnetic field. This causes the salt pill or refrigerant to cool, and it is able to suck heat from an object, e.g., the sensor, to be cooled. Current has to be fed into and out of the magnets from a dissipative power supply at the ambient temperature of the spacecraft. The current leads that link the magnets to the power supply inevitably conduct a significant amount of heat into the colder regions of the supporting cryostat, resulting in the need for larger, heavier, and more powerful supporting refrigerators. The aim of this project was to design and construct high-temperature superconductor (HTS) leads from YBCO (vttrium barium copper oxide) composite conductors to reduce the heat load significantly in the temperature regime below the critical temperature of YBCO.

The magnet lead does not have to support current in the event that the YBCO ceases to be superconducting. Customarily, a normal metal conductor in parallel with the YBCO is a necessary part of the lead structure to allow for this upset condition; however, for this application, the normal metal can be dispensed with.

Amorphous silicon dioxide is deposited directly onto the surface of YBCO, which resides on a flexible substrate. The silicon dioxide protects the YBCO from chemically reacting with atmospheric water and carbon dioxide, thus preserving the superconducting properties of the YBCO. The customary protective coating for flexible YBCO conductors is silver or a silver/gold alloy, which conducts heat many orders of magnitude better than SiO₂ and so limits the use of such a composite conductor for passing current across a thermal gradient with as little flow of heat as possible to make an efficient current lead. By protecting YBCO on a flexible substrate of low thermal conductivity with

SiO₂, a thermally efficient and flexible current lead can be fabricated. The technology is also applicable to current leads for 4 K superconducting electronics current biasing.

A commercially available thin-film YBCO composite tape conductor is first stripped of its protective silver coating. It is then mounted on a jig that holds the sample flat and acts as a heat sink. Silicon dioxide is then deposited onto the YBCO to a thickness of about 1 micron using PECVD (plasma-enhanced chemical vapor deposition), without heating the YBCO to the point where degradation occurs.

Since SiO_2 can have good high-frequency electrical properties, it can be used to coat YBCO cable structures used to feed RF signals across temperature gradients. The prime embodiment concerns the conduction of DC current across the cryogenic temperature gradient. The coating is hard and electrically insulating, but flexible.

This work was done by Daniel Johannes and Robert Webber of Hypes for Goddard Space Flight Center. Further information is contained in a TSP (see page 1). GSC-16732-1