Cold Sintering Creates New Avenues for Advanced Composites Manufacture

Engineers often want to combine many types of materials into a composite architecture to achieve specific performances in devices, from electronics to biomedical implants. However, material compatibility issues that arise during manufacturing can limit practical realization of these composites.

Sintering — a common technique to engineer composites — transforms constituent material powders, with heat, into a dense layer or solid body. Sintering traditionally occurs at extremely high temperatures near, but below, the material's melting point (*e.g.,* most ceramics require temperatures exceeding 1,000°C). However, such high temperatures produce undesired outcomes, such as the melting of metals, decomposition of polymeric materials, and interfacial reactions.

Material incompatibilities make combining metals, plastics, and ceramics unfeasible in many applications. Thus, engineers are motivated to search for better ways to sinter diverse materials at lower temperatures.

Researchers at the Center for Dielectrics and Piezoelectrics (CDP) — a National Science Foundation Industry-University Cooperative Research Center (NSF IUCRC) involving North Carolina State Univ. and Pennsylvania State Univ. — have discovered a technique to sinter materials at low temperatures to form new materials with densities that approach the theoretical limit of zero porosity. This technology, called the cold sintering process (CSP), offers new opportunities for integrating previously incompatible materials and drastically lowers the energy and time required in the sintering process.

▲ Cold sintering creates composite materials with lower energy costs, such as Li_2MoO_4 capacitor structures on nickel foil, which is sintered at 100 MPa for 15 min at 120°C.

Clive Randall of Penn State, the lead researcher on CSP, says, "Not only is CSP a low-temperature process (room temperature up to 300°C), but we are also densifying some materials to over 95% of their theoretical density in 15 minutes. We can now make a ceramic faster than you can bake a pizza, and at lower temperatures."

To perform CSP, operators wet ceramic powder with a few drops of water or an acid solution, which partially dissolves the solid surfaces of the particles at the particle-particle interfaces. The temperature and pressure are increased (to 100–300°C and 100–350 MPa) to trigger an initial non-equilibrium thermodynamic densification process: First, the solid particles rearrange. Then, elemental constituents from the solid particles dissolve and diffuse through the supersaturated liquid phase. Next, particles densify into a solid body and reprecipitate. Locally high pressures near the particle interfaces mimic typical hydrothermal crystal growth conditions, which are important in geological mineral formation.

"I see the cold sintering process as a continuum of different significant challenges," Randall says. "In some systems, it's so easy, you don't need pressure. In others, you do. In some, you need to use nanoparticles. In others, you can get away with a mixture of nanoparticles and larger particles. It really all depends on the systems and chemistries you are talking about, the inherent transport mechanisms in play, and the structure-property relationships." Researchers at the CDP seek to explore the fundamental mechanisms and model the processes that drive cold sintering.

The team is building a library of the precise techniques required to use CSP on various material systems, such as ceramic-ceramic composites, ceramic-nanoparticle composites, ceramic-metals, and ceramic-polymers. They have verified 50 processes to date. They are also extending the scope of research to a broad range of inorganic and organic-inorganic dielectrics that are of interest to the dielectrics and piezoelectrics industry.

Richard Clark, senior technical specialist for Morgan Advanced Materials (a large manufacturing corporation specializing in advanced materials science and engineering of ceramics, carbon, and composites), believes that cold sintering is a step change for ceramics materials processing. Lower temperatures reduce energy costs of production as well as allow for new combinations of materials and functionalities. Applications for cold sintering include:

- reinforced ceramic composites for toughened refractories
- solid-state ionically conducting ceramic electrolytes for next-generation lithium ion batteries
- ceramics with embedded sensors for use as medical implants.

"The savings in energy costs could be worth billions to industry, if the process could be ramped up to factory-floor scale," says Clark. CEP

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