Investigation of Ni/Al₂O₃ nacre-like composite through hot pressing of freeze-cast foams

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Why mimicking nacre ?

The development of materials that combine high strength and toughness is crucial for structural applications where catastrophic failures can not be tolerated. Despite being mainly composed of brittle materials, nacre, found in seashells, exhibits remarkable damage-tolerant properties. It is composed at 95vol.% of aragonite platelets that are aligned on a long-range order and bonded by a thin organic layer. For this reason, the structure of nacre is referred to as a brick-and-mortar structure. Its hierarchical structure exhibits several toughening mechanisms which enable its toughness to be as high as three orders of magnitude (in energy terms) greater than either its components. Hence, it makes natural nacre an ideal case study to achieve damage-tolerant materials.

Conclusions

Objectives

This project investigated the development of a nacre-like composite made of micron-size alumina platelets coated with nickel to achieve a damage-tolerant composite that can withstand elevated temperature. The goal of using coated platelets was to achieve a brick-and-mortar structure where the alumina platelets act as the bricks, to provide strength to the composite, while the nickel acts as the mortar, to toughen the structure. The development of the composite emphasizes the two milestones:

- Investigation of the toughening mechanism in a Ni-Al₂O₃ brick-and-mortar structure

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Process

Electroless nickel (EN) plating

This method enables the deposition of nickel onto non-conductive surfaces such as alumina. The deposition was realised onto alumina platelets to provide a uniform and continuous distribution of nickel throughout the composite. It was achieved by immersing the platelets into three successive chemical solutions, respectively for sensitisation, activation and deposition. The sensitisation and activation steps make their surface catalytical thanks to the adsorption of tin and palladium ions. The last solution contains nickel ions and a reducing agent that promotes their reduction and leads to the deposition of nickel onto the alumina platelets.

Freeze-casting

Freeze casting consists of freezing a liquid suspension, where the solidified liquid is then sublimated to obtain a porous scaffold that is sintered for densification. The liquid suspension was realised with water as solvent because it leads to a lamellar structure which is convenient for further pressing. In addition, the lamellar growth enables to align the nickel coated alumina platelets trapped in-between the ice lamellae. Due to the low amount of nickel deposited via the electroless nickel deposition, the nickel content of the final composite was further increased by adding nickel oxide nanoparticles into the liquid suspension. The solidification was forced by placing the liquid suspension on top of a cooled copper plate at ~20°C for 2h at a cooling rate of 10°C/min. As the orientation of the ice lamellae is defined upon nucleation which occurs randomly on the cold surface, a wedge was used to constrain the ice to grow parallel to each other. The utilisation of a wedge enables to confine the nucleation and affects the thermal gradient during solidification which lead to a bi-directional scaffold. Solidification was performed in a freeze-dryer where the pressure was decreased to 1.10⁻⁶ atm while the temperature was maintained at ~50°C for 48h.

Reduction/sintering

After freeze-casting, the constituents are weakly bonded together as they were simply trapped together in-between ice-lamellae. The structure is therefore sintered to bond the particle together which provides its structural stability. Sintering was realised in a hydrogen furnace to reduce the nickel oxide at 1050°C for 4 hours.

Shaping

The samples were shaped in a cylinder with the composite lamellae perpendicular to its longitudinal axis. This step was realised manually by using sandpapers. It enabled to press the cylinder while keeping the lamellar alignment and therefore the alignment of the platelets.

Hot pressing

The macroporosity of the structure was removed by pressing the cylinder along its axis to achieve a brick-and-mortar structure. Pressing was realised under a pressure of 60MPa for 2 hours at 1200°C. During this final step, sintering between the components gave the composite its optimal mechanical properties.

Results

Summary

- 3 compositions were realised: alumina with 1vol.%, 10vol.% and 30vol.% of nickel.
- Addition of nickel oxide in the slurry leads to nickel agglomerates after reduction that gets larger as the nickel content increases.
- Inter-platelets spacing leads to high porosity (~30%) for composite with low nickel content (1vol.% and 5vol.%).
- Increasing the nickel content to 30vol.% reduces the porosity to 16% and provides a continuous mortar layer.
- Hot pressing further improves the platelets alignment.
- Nickel enables to bond alumina at low temperature (1200°C).
- Large agglomerates in the freeze-cast structure leads to inhomogeneous mortar thickness.

Discussion

- Toughening mechanisms qualitatively observed in a composite with 10vol.% of nickel.
- Crack path follows the alignment of the platelets at high energy while it is deviated at low energy.
- Platelets pull-out leads to tortuous crack path.
- Friction between platelets and the mortar lowers the crack-driving force.
- Nickel coating provides asperities on the surface of each platelet that toughen and strengthen the composite as it creates interlamellae bridges similar to natural nacre.

Acknowledgements

I acknowledge Prof. David Dunand for welcoming me in his research group and for his precious guidance throughout the development of this project. I especially thank Prof. Andreas Mortensen for this opportunity and his support along this work. Many thanks to Eric Maire, the Mateis group, the Dunand group and the thermoelectrics group. Thanks to Eric Maire, the Mateis group, the Dunand group and the thermoelectrics group. Thanks to Eric Maire, the Mateis group, the Dunand group and the thermoelectrics group.

Reference