Plasma-based surface treatment for improving joint strength of joined ceramics and ceramic matrix composites – PhD Thesis summary

Joining ceramics enables the manufacturing of parts able to face challenging conditions such as high temperatures and corrosive operational environments.

To manufacture a reliable product, promoting optimal adhesion between the parts to be joined and the joining material is critical. This can be promoted by a preliminary preparation of the surface and in particular by modifying the surface texture and chemistry.

Surface texturing of ceramics can be costly, time-consuming, and difficult with traditional techniques like mechanical machining and wet etching because of their hardness, brittleness, and chemical inertness. These limits can be overcome by non-contact surface texturing techniques based on plasma and laser, promising solutions that are recognized as promising, but more research is needed.

This thesis aimed to explore and identify new plasma-based solutions for improving the joint strength of joined ceramics and ceramic matrix composites (CMCs) by inducing a new surface texture able to promote interlocking. Plasma treatments are well-known in the preparation of polymeric surfaces and the manufacturing of semiconductors, but little information is available on their use for increasing the resistance of the joints of ceramics. Therefore, this work gives a contribution to extending the available knowledge and it pays special attention to identifying treatments that can be implemented at an industrial scale.

A commercial corona plasma treater was used as a solution for increasing the joint strength of similar joints of bulk silicon carbide (SiC) bonded with itself using an epoxy adhesive. Such treatment is widely known in the plastic industry, but it was never investigated for improving glue adhesion on SiC. Characterization of the surface before and after the treatment was carried out and it was observed the formation of a corona-induced cauliflower-like silica layer on the surface. Both surface chemistry and texture were modified. Mechanical tests (SLO compressive test) found an increase in joint strength after the corona treatment, from 61.5 ± 5.0 MPa to 68.8 ± 2.3 MPa. Furthermore, the failure mode changed from adhesive to cohesive, pointing out a better adhesion of the glue and the uniformity of the treatment.

For CMCs, the difference in terms of the etching resistance of each constituent was exploited to manufacture a brush-like superficial texture beneficial for enhancing the infiltration of the braze and promoting an interlocking effect.

Fluorine-based Reactive Ion Etching (RIE) is a low-pressure plasma technique extensively used for manufacturing semiconductor devices, but it has never been proposed as a pre-joining treatment for SiC/SiC composites. In this work, an RIE plasma was used to induce a selective removal of fibers in order to obtain the brush-like texture. Several plasma conditions were considered and, after preliminary observations, one of them (CF_4, 20 sscm, 200 W, 30 min) was selected for assessing the effects of the treatment on the joint strength. A commercially available AgCuTi braze was used as the joining material. The joint strength of joined SiC/SiC was recorded to increase by 55% after the treatment (SLO compressive test).

The use of a commercial air-fed Atmospheric Pressure Plasma Jet (APPJ) was evaluated to induce selective removal of fibers and, therefore, manufacturing a brush-like texture on carbon-fiber reinforced carbon (C/C) and carbon-fiber reinforced silicon carbide (C/SiC). In both cases, the treatment resulted in the formation of a brush-like texture since fibers were removed preferentially. This was more remarkable for C/SiC because of the strong difference in terms of oxidation resistance between the carbon fibers and the silicon carbide matrix. The novel texture promoted the infiltration of the braze (TiCuNi system for C/C and AgCuTi for C/SiC) and the consequent interlocking.

Similar joints of C/C were affected by an intense depletion of joining material given by the presence of existing large and interconnected porosities in the composites, while for dissimilar joints copper-C/C the joint strength was higher for APPJ-treated joined samples (22.8 ± 6.5 MPa) then for the untreated (9.5 ± 7.4 MPa). The joint strength of C/SiC, when joined with itself, increased from 45.5 ± 0.6 MPa to 65.8 ± 2.5 MPa after the APPJ treatment. All values were collected through SLO compressive test.

All the plasma treatments investigated in this thesis proved to be promising as pre-treatment before joining in order to improve the final joint strength and the results set the ground for new research on the topic while contributing to generating new knowledge in a little-explored field that is expected to be critical for unlocking novel technologies in next years.