Chapter 7

Conclusions and Future Prospects

7.1 Conclusions

Advanced materials are the materials which are engineered for outstanding performance, typically achieved through sophisticated manufacturing techniques. Reusable launch vehicles (RLVs) encounter significant aerodynamic heating at the stagnation point, attributed to high Mach numbers (>5). The mitigation of this heat is achieved through thermal protection systems. Ceramic matrix composites (CMCs) represent a novel category of lightweight advanced materials known for retaining their properties even at temperatures exceeding 1000ºC.

In numerous instances, the application demands CMC hardware with either substantial geometric dimensions or intricate shapes. Traditional methods such as welding, extrusion moulding, and forging prove impractical for realizing CMC components. Furthermore, the difficulties extend to the reclamation of damaged parts and the identification of faults, especially in complex shapes. To facilitate processing, larger components are divided into several segments, allowing for easier fabrication and quality inspection, and enhancing safety. Therefore, the need for suitable joining technology for integrating ceramics-to-ceramics or ceramics-to-metals arises to accomplish the specified requirements. The major conclusions drawn from the present study are as follows:

(1) Joining of C/SiC to C103 alloy

- ❖ C/SiC and niobium alloy (C103) were successfully joined by Ti-containing Ticusil® and Cusil® active braze alloy.
- ❖ According to the titanium concentration in the braze alloy, the interfaces of C/SiC–C103 joints exhibited an observable reaction zone with a thickness ranging from

approximately 1 to 3 μm, mainly composed of titanium carbide near the C/SiC substrate.

- ❖ The interphase comprises TiC, Nb_2C, and Ti_5Si_3 as newly formed phases, as characterised by XRD and EDS techniques. Also, the titanium content in braze alloy governs the concentration of these phases at the interface. Due to the fragility of these phases, a low concentration is necessary to obtain maximum binding strength.
- ❖ The optimisation study suggested that high temperature (838°C), intermediate reaction time (34 min), and low cooling rate (1°C/min) are desirable for obtaining the highest LSS value of 12 MPa for Ticusil-based joint.
- ❖ A maximum average LSS value of 16 MPa was reached for the Cusil-based joint when the brazing temperature of 840°C, a reaction time of 10 min, and a cooling rate of 5°C/min were maintained.

(2) Joining of C/SiC composites

- ❖ A novel carbon nanotubes (CNTs) reinforced Ni-Si-CNTs composite filler was developed to braze the C/SiC with C/SiC with improved mechanical properties. Maximum LSS of 21 MPa achieved for Ni-Si-CNTs alloy containing 10 vol.% CNTs.
- ❖ The LSS value was ~90% of the interlaminar shear strength of C/SiC and 147% higher than the joint without CNTs addition.
- ❖ The joining of C/SiC without CNTs suffers from huge voids and cracks formation at the interface due to a significant difference in CTE between the C/SiC.
- ❖ The addition of CNTs provides additional reinforcement to the interlayer and decreases the overall CTE value leading to lesser residual stresses while cooling.
- ❖ The filler converts to an interlayer comprising high-temperature stable phases i.e., Ni_2Si, Ni_3Si_2, and β-SiC.
- ❖ The loading of CNTs above 10 vol. % significantly lowers the bond strength due to excessive agglomerate formation emerging as the weakest point of the interlayer.

(3) Joining of monolith SiC ceramics

- ❖ A novel Mo-containing Ni-Si-Mo composite filler was designed to join monolith SiC ceramics by a brazing process.
- ❖ The Mo-addition decreases the overall CTE value of the filler and consumes the precipitated graphite formed during the brazing process.

- Mo content of 8 at.% rendered a state of thermodynamic equilibrium with SiC without the presence of graphite at the interface.
- The CTE value decreases from 13.6 to 5.4×10^{-6}/°C due to 8 at.% Mo addition into the Ni-Si filler.
- Maximum LSS of 107 MPa was achieved and the interphase was composed of thermodynamically high-temperature stable Ni-based (e.g., δ-Ni_2Si, Ni_3Si_2) and Mo-based (e.g., Mo_2C, Ni_3Mo_3C) phases.
- Mo loading exceeding 12 at.%, demonstrates a significant increase in unit cell volume, leading to the development of a thicker interphase and a non-homogeneous distribution of the phases causing low bond strength.

The maximum expected service temperature for the Ticusil/Cusil-based C/SiC-C103 joints is 600°C. On the other hand, Ni-Si-CNT and Ni-Si-Mo-based joints are expected to perform satisfactorily upto 900°C.

7.2 Future prospects

- The Ceramic-based joints are intended for high-temperature applications. Therefore, the high-temperature joint strength should be evaluated considering the end application.
- The topology of the joining surfaces is very crucial. Therefore the effect of nail-like structure on bond strength, should be explored.
- The next-generation ceramic materials are ultra-high temperature ceramics (UHTCs) such as (Ti, Zr, Hf)C, (Ti, Zr, Hf)B_2, and so on. As a result, emphasis is required for the joining of these materials to themselves or metals.
- Modelling of residual stresses at the joining interface and measurement by characterisation techniques.
- In the modelling of interface joints, it is crucial to consider compound formation at the interface under realistic conditions. Existing investigations often assume room temperature conditions, which deviate from the actual scenario where compounds form at the joining temperature and undergo subsequent cooling to room temperature.
- Future work should be oriented towards the realization of joints having minimum thermal residual stresses. Studies should also be extended to minimize the CTE to the best possible value using negative CTE compounds.

- ❖ The spreading of molten liquid on a ceramic base at joining temperatures involves chemical reactions and diffusions, a phenomenon that can be comprehended through theoretical calculations on the energetics of molten metal-ceramic interfaces using statistical mechanics. Further research in this area is needed to account for these factors and achieve optimal joints.
- ❖ High-entropy alloys (HEAs) are a new class of materials with excellent properties i.e., high entropy of mixing, sluggish diffusion, etc, which cause exceptional characteristics such as high strength at elevated temperature, excellent oxidation and corrosion resistance, and superior resistance to radiation damage. The use of high entropy alloy (HEAs) as brazing filler, has a high potential to produce a good quality joint for extreme applications.