FABRICATION OF CERAMIC MATRIX COMPOSITES BY LIQUID SILICON INFILTRATION (LSI)

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Liquid Silicon Infiltration (LSI) – description

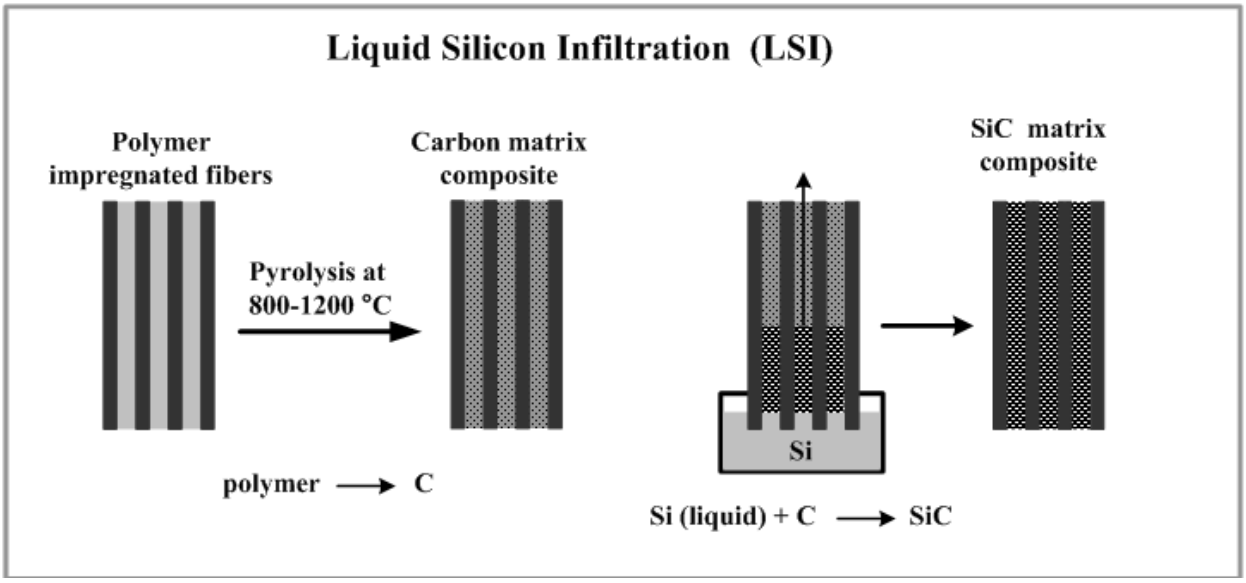
Liquid Silicon Infiltration (LSI) process is a type of Reactive Melt Infiltration (RMI) technique, in which the ceramic matrix forms as a result of chemical interaction between the liquid metal infiltrated into a porous reinforcing preform and the substance (either solid or gaseous) surrounding the melt.

Liquid Silicon Infiltration (LSI) is used for fabrication of silicon carbide (SiC) matrix composites. The process involves infiltration of carbon (C) microporous preform with molten silicon (Si) at a temperature exceeding its melting point 2577°F (1414°C).
The liquid silicon wets the surface of the carbon preform. The melt soaks into the porous structure driven by the capillary forces. The melt reacts with carbon forming silicon carbide according to the reaction:

Si(liquid) + C(solid) → SiC(solid)

SiC produced in the reaction fills the preform pores and forms the ceramic matrix. Since the molar volume of SiC is less than the sum of the molar volumes of silicon and carbon by 23%, the soaking of liquid silicon continues in course of the formation of silicon carbide. The initial pore volume fraction providing complete conversion of carbon into silicon carbide is 0.562. If the initial pore volume fraction is lower than 0.562 the infiltration results in entrapping residual free silicon. Commonly at least 5% of residual free silicon is left in silicon carbide matrix.

The porous preform may be fabricated by either pyrolysis of a polymerized resin or by Chemical Vapor Infiltration (CVI). The preform microstructure is important for complete infiltration. Large pores helps to obtain a complete infiltration but may result in non-complete chemical interaction and formation of a structure with high residual free silicon and unreacted carbon. Small preform pores results in more complete chemical reaction but in non-complete infiltration due to the blockage (chock-off) of the infiltration channels.



In contrast to the composites fabricated by Polymer Infiltration and Pyrolysis (PIP) and Chemical Vapor Infiltration (CVI) ceramic matrices formed by Liquid Silicon Infiltration are fully dense (have zero or low residual porosity).

The infiltrated at high temperature molten silicon is chemically active and may not only react with the carbon porous preform but also attack the reinforcing phase (SiC or C fibers, whiskers, or particles). A protective barrier coating (interphase) of SiC, C or Si_3N_4 prevents the damage of the fibers by the melt. The barrier coatings are applied over debonding coatings (pyrolytic carbon (C) and hexagonal boron nitride (BN)). The interphases may be deposited by Chemical Vapor Infiltration (CVI). The protective barrier from pyrolytic carbon is formed by Polymer Infiltration and Pyrolysis (PIP).

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Liquid Silicon Infiltration (LSI) process

- **Application of Interphases**. A thin (commonly 0.1-1 μm) layer of a debonding phase (pyrolytic carbon (C) or hexagonal boron nitride (BN)) is deposited on the fiber surface by Chemical Vapor Infiltration (CVI) method. In addition to this the fibers are protected from the highly reactive liquid silicon by a barrier coating (commonly SiC). The interphases are deposited by Chemical Vapor Infiltration (CVI).
- **Fabrication of the prepreg**. The reinforcing fibers (tow, tape, weave) are impregnated with a resin and then dried or cured to B-stage (partial curing). The resin contains carbon, which further will react with molten silicon.
- **Lay-up**. The prepreg is shaped by a tooling (mold).

- **Molding**. The laid-up prepreg is molded. Various molding methods may be used. In the bag molding a rigid lower mold is combined with a flexible upper mold (bag), which is pressed against the prepreg by either atmospheric pressure (vacuum bag mold) or increased air pressure (gas pressure bag mold). The pressurized preform is cured in an autoclave. A combination of a pressure with an increased temperature may also be achieved in compression molding.
- **Pyrolysis**. Pyrolytic decomposition of the preceramic polymer is performed in the atmosphere of Argon at a temperature in the range 1472-2192°F (800-1200°C). Volatile products are released as a result of pyrolysis forming a porous carbon structure.
- **Primary machining**. This operation may be performed after the steps of molding and/or pyrolysis.
- **Infiltration of the porous prepreg with Liquid Silicon**. The prepreg is immersed into a furnace with molten silicon where its porous carbon structure is infiltrated with the melt. The infiltration process is driven by the capillary forces. Liquid silicon reacts with carbon forming in situ silicon carbide matrix.
- Final machining.

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