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Space engineering — Structural materials handbook

Part 7: Thermal and environmental integrity, manufacturing aspects, in-orbit and health monitoring, soft materials, hybrid materials and nanotechnologies

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European Foreword

This document (CEN/TR 17603-32-07:2022) has been prepared by Technical Committee CEN/CLC/JTC 5 “Space”, the secretariat of which is held by DIN.

It is highlighted that this technical report does not contain any requirement but only collection of data or descriptions and guidelines about how to organize and perform the work in support of EN 16603-32.

This Technical report (CEN/TR 17603-32-07:2022) originates from ECSS-E-HB-32-20 Part 7A.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This document has been developed to cover specifically space systems and has therefore precedence over any TR covering the same scope but with a wider domain of applicability (e.g.: aerospace).

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Introduction

The Structural materials handbook is published in 8 Parts.

A glossary of terms, definitions and abbreviated terms for these handbooks is contained in Part 8.

The parts are as follows:

TR 17603-32-01	Part 1	Overview and material properties and applications	Clauses 1 - 9
TR 17603-32-02	Part 2	Design calculation methods and general design aspects	Clauses 10 - 22
TR 17603-32-03	Part 3	Load transfer and design of joints and design of structures	Clauses 23 - 32
TR 17603-32-04	Part 4	Integrity control, verification guidelines and manufacturing	Clauses 33 - 45
TR 17603-32-05	Part 5	New advanced materials, advanced metallic materials, general design aspects and load transfer and design of joints	Clauses 46 - 63
TR 17603-32-06	Part 6	Fracture and material modelling, case studies and design and integrity control and inspection	Clauses 64 - 81
TR 17603-32-07	Part 7	Thermal and environmental integrity, manufacturing aspects, in-orbit and health monitoring, soft materials, hybrid materials and nanotechnoligies	Clauses 82 - 107
TR 17603-32-08	Part 8	Glossary	

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82 Thermal behaviour

82.1 Introduction

82.1.1 General

The two principal issues in the behaviour of high-temperature materials are the:

- physical response to thermal loads, i.e. thermal cycling and thermal shock.
- physical properties, i.e. thermal conductivity and diffusivity, specific heat capacities, emissivity and surface catalyticity.

82.1.2 Physical response

Information on physical response shows the temperature ranges to which different materials can be applied and the level of microstructural changes that can occur. This is viewed in conjunction with the effects of thermal loads which are likely to be compounded by:

- mechanical loading, [See: Chapter 83], and
- operating environments, [See: Chapter 85].

Any modification to the material's microstructure alters the physical properties.

82.1.3 Physical properties

Physical properties are used to establish the amount of heat energy that a structural design can absorb. Thermal conductivity is a non-linear parameter whose value is temperature dependent.

Thermal cycling is divided into two major regimes:

- Low Range: Cryogenic up to 300°C. For:
  - magnesium,
  - aluminium, and
  - glass composites.
- High Range: Above 300°C. For propulsion system materials and thermo-structural designs, i.e.
  - copper,
  - titanium,

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— nickel,
— GCMC,
— CMC, and
— C-C

Where cryogenic LH/LOX fuels are used, thermal cycling can extend from -253°C to +800°C.

The thermal behaviour of materials is influenced by:

- plastic behaviour (MMC),
- thermal strain hysteresis (MMC and CMC),
- microvoidage and fibre-matrix interface damage (MMC),
- porosity (CMC),
- microcracking (CMC).

82.2 MMC: Thermal cycling

82.2.1 General

Thermal cycling damage is more likely to occur with, Ref. [82-4]:

- significant CTE mismatch between reinforcement and matrix,
- high residual stresses within the matrix phase,
- over-extension of upper temperature capabilities of matrix or fibre,
- repeated thermal cycling.

Reinforcement of metals increases the upper service temperature for very short life applications by limiting gross creep. For repeated thermal cycling and longer life requirements, accumulating changes in matrix microstructure inhibit the upper service temperature. Some published work shows that over-extension of the temperature limits causes degradation, e.g. an accumulation of micro-damage associated with the matrix-reinforcement interfaces (microvoids or cracks at the interfaces) often leading to interfacial debonding.

Thermal cycling a MMC under constant mechanical load can cause a ratcheting effect. This shakedown phenomenon results in small increments in permanent strain on each thermal cycle, Ref. [82-1].

Information on gross changes in mechanical performance due to thermal cycling is provided.

[See: Chapter 84 for modifications to CTE behaviour from lower thermal cycling regimes]

82.2.2 Magnesium-carbon fibre composites

Combining magnesium with UHM carbon fibres is attractive for dimensionally stable structures. In these, permanent residual strain is induced on the first thermal cycle, Ref. [82-2], but not subsequently.

Important for designing dimensionally stable mirrors, for example.

Similar residual strains can be induced in aluminium composites with UHM fibres, e.g. P100.

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82.2.3 Aluminium-carbon fibre composites

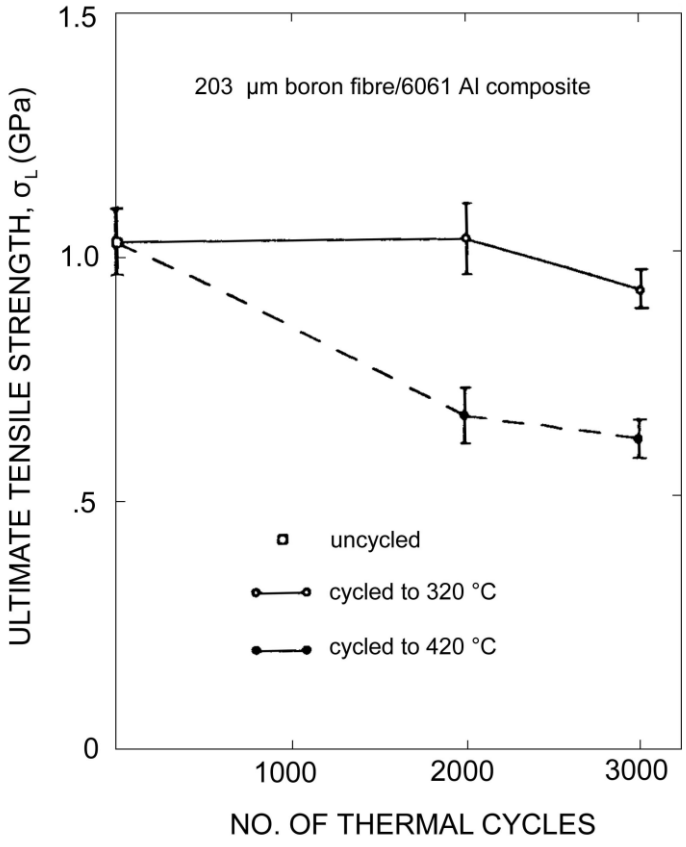
The combination of carbon fibres and aluminium causes a brittle aluminium carbide interface due to chemical reaction between the two. Thermal cycling to 350°C can result in severe losses in mechanical performance, Ref. [82-3]. Special attention should be given to the interface with C/Al composites if they are to retain their integrity.

82.2.4 Aluminium-boron filament composites

Severe damage from thermal cycling was seen in boron fibre-reinforced 6061 aluminium alloy in cycling to 320°C or 420°C, Ref. [82-4]. Gross failure of the fibre-matrix interface occurred throughout the composite.

The unidirectional strength properties reduced significantly, as shown in Figure 82.2.1, Ref. [82-4]. The transverse 90° properties were effectively destroyed giving gross embrittlement. The work showed that the peak temperatures were excessive for prolonged composite use as the composite was losing integrity.

The base alloy is not normally used above 120°C.



RT UTS of 203µm boron fibre/6061 Al composites.

Thermally cycled between RT and Tmax (320°C and 420°C)

Figure 82.2-1 - MMC thermal cycling: Effect on UTS for boron/Al composite

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82.2.5 Titanium-silicon carbide filament composites

Higher performance titanium alloys can be used at temperatures up to 600°C. Prior to replacement by Beta 21S (Timet 21), Ti-15-3-3 was a β-alloy studied widely in the USA, [See also Chapter 47].

Quasi-isotropic SCS6/Ti-15-3-3 composites were exposed to differing thermo-cyclic regimes, i.e.:

- 149°C to 427°C,
- 316°C to 649°C,
- isothermal fatigue at 649°C.

From 149°C to 427°C, the sequence observed was, Ref. [82-5]:

- after 500 cycles, the reaction zone (interface) between fibre and matrix changed in appearance,
- radial cracks grew through the reaction zone,
- at 1500 cycles, cracks initiated on the edge of the specimen,
- edge cracks were arrested at fibres,
- after 16750 cycles, the composite was still intact.

The conclusion was that the damage was very minor and the composite was capable of sustaining many cycles at temperatures up to 427°C.

From 316°C to 649°C, [0/±45/90]s laminates exhibited significant damage after several hundred cycles, Ref. [82-6], such as:

- significant fibre-matrix debonding,
- ply-to-ply separation (delamination) in the 45° and 90° plies,
- contact or close proximity of fibres increased the occurrence of cracking.

82.2.6 Superalloy (FeCrAlY) composites

Thermal cycling tests have been conducted on FP alumina/FeCrAlY and W-ThO_2/FeCrAlY composites, [See: Chapter 48]. The constituents were selected to minimise the CTE mismatch between matrix and reinforcement, Ref. [82-7]. Thermal cycling was taken to 1100°C for up to 200 cycles. Severe microstructural damage occurred, which emphasises the difficulties in producing MMC materials that are expected to sustain large changes in temperature.

82.3 CMC: Thermal cycling

Ceramic composites (CMCs) differ from MMC materials in not having a plastically deformable matrix. Microcracks, created in the matrix on cooling, are the internal stress relief mechanism. Microcracks are tolerated in CMCs as a means of gaining a benign fracture behaviour.

Thermal cycling does not contribute significantly to accumulated internal damage in CMCs, unless the thermal limit of the fibre is exceeded. Thermal cycling involves incremental heating throughout the specimen at a fairly constant rate.

Ceramics are more likely to undergo rapid high temperature excursions with uneven through-thickness heating. Thermal shock is therefore a more severe test for CMCs, particularly as the low overall conductivity gives rise to large temperature gradients, [See: 82.5]

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82.4 MMC: Thermal shock

82.4.1 General

Thermal shock occurs in propulsion systems with the ignition of high energy fuels. The high thermal conductivity of metals assists in dissipating heat away from the immediate surface.

82.4.2 Metal alloys

Metallic materials applied to propulsion systems and experiencing thermal shock include:

- copper alloys (including ODS alloys), [See: Chapter 45].
- nickel alloys, [See: Chapter 48].
- refractory metals, e.g. niobium and tantalum, [See: Chapter 50].

All have shown acceptable thermal shock resistance.

For short duration firings, creep can occur in combustion chambers, e.g. Vulcain, showing the combined effects of temperature and pressure to be severe.

82.4.3 MMC

TMC, such as SiC/Ti composites, are destined for gas turbine applications (blisks) and feature significantly in the NASP programme, [See: Chapter 71]. Blisks are integrally-formed turbine blade and disc components.

Actual applications of MMC materials are unknown.

82.4.4 Intermetallics

Titanium aluminides (TiAls) are also under evaluation for gas turbine applications, [See: Chapter 49].

82.5 CMC: Thermal shock

82.5.1 General

Continuous fibre GCMC, CMC and C-C materials have shown a remarkable resistance to severe thermal shock, Ref. [82-8].

These materials do not undergo the single crack, catastrophic mode of failure of monolithic ceramics.

The materials can sustain a high degree of microcracking whilst still retaining their basic integrity.

This is true even when the fibres have undergone severe thermal degradation, as with SiC fibres taken to 1700°C. Their mechanical strengths are lowered by repeated thermal shocks, but continue to perform a thermo-structural function. They are in effect a form of rigidised fibre insulation material.

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82.5.2 SiC-SiC composites

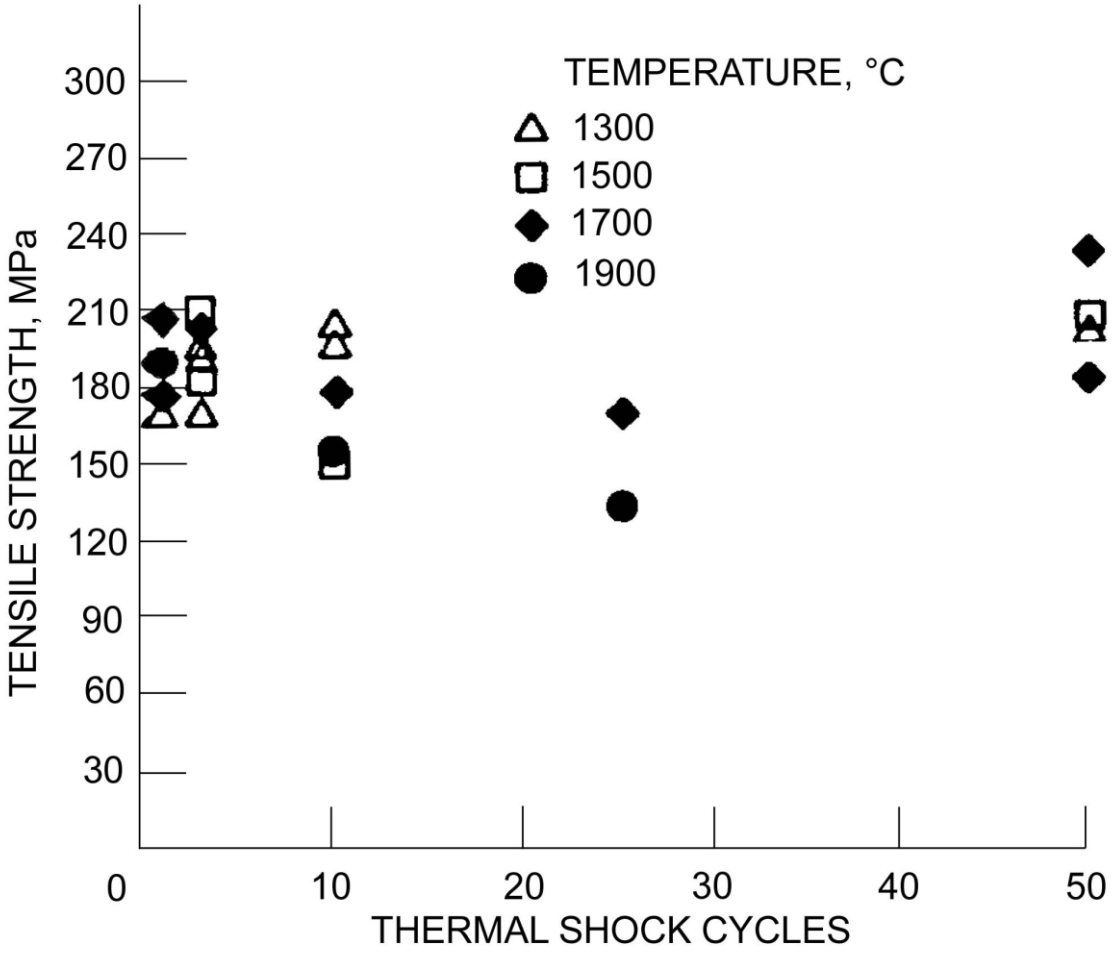
As an example of their resistance, SiC-SiC composites have undergone thermal shock conditioning more severe than that seen in the current Space Shuttle Main Engine (SSME) turbopump, Ref. [82-9].

Figure 82.5.1 shows the strength retention of SiC-SiC after exposures to 1900°C, Ref. [82-9].

At 1900°C, material erosion produced a 40% cross-sectional reduction after 25 cycles. Little or no decrease in tensile strength for thermal shock up to 1700°C was seen. The testing comprised of a hydrogen-oxygen rocket engine firing in one second pulses.

Other methods used to demonstrate shock resistance are:

- using a high intensity electron beam, Ref. [82-10], or
- plunging white-hot CMC specimens into water.



Material: Nicalon/SiC

Figure 82.5-1 - Thermal shock: Retained RT tensile strength for SiC-SiC CMC

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82.6 MMC: Thermal conductivity

82.6.1 General

Thermal conductivity of MMC materials varies with temperature.

Thermal diffusivity is the parameter measured directly and thermal conductivity calculated by:

$$\lambda = \alpha \rho C_p \qquad [82.6\text{-}1]$$

where:

λ = *thermal conductivity*

α = *thermal diffusivity*

ρ = *material density*

C_p = *specific heat capacity*

82.6.2 Thermal diffusivity measurement

Accurate measurement of thermal diffusivity is best achieved by a laser flash method, introducing a precisely quantified amount of heat energy, Ref. [82-11]. This said, published data on the diffusivity of structural materials is often inconsistent and ambiguous. With the anisotropic nature of composites, diffusivity is influenced by factors, including:

- reinforcement orientation,
- alloy and reinforcement composition,
- voidage,
- interface materials.

82.6.3 Effect of material composition

82.6.3.1 General

Introducing ceramic reinforcing phases into metallic materials usually lowers thermal conductivity. There are some exceptions, e.g.

- titanium has very low thermal conductivity in comparison with aluminium and copper,
- crystalline silicon carbide and carbon have good conductivities with a reduced lowering effect when compared with alumina,
- highly graphitised reinforcements have excellent conductivity.

Where high thermal conductivity is essential, a number of specialised composites have appeared which retain this characteristic at medium to high temperatures.

Copper has excellent thermal conductivity, but modest strength characteristics and a high density. It is used sparingly in some propulsion systems, e.g. the Vulcain engine for Ariane 5, but copper composites offer better structural capabilities in conjunction with high conductivity, Ref. [82-11].

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[See: Chapter 45]

82.6.3.2 Particulate reinforcements

The effect on thermal conductivity of particulate additions to titanium is shown in Figure 82.6.1, Ref. [82-12].

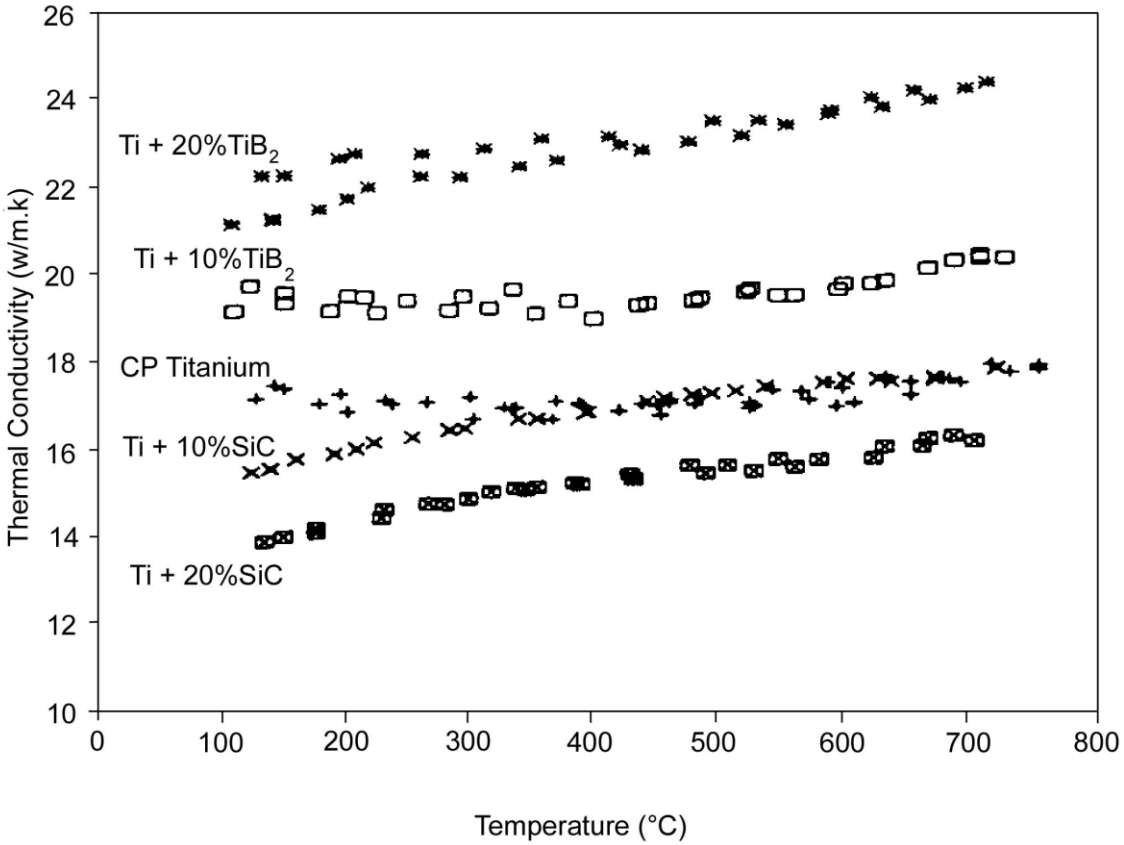


Figure 82.6-1 - Thermal conductivity: Unreinforced titanium compared with Ti-SiC and Ti-TiB2 particulate reinforced composites

82.6.4 Modelling

Prediction of thermal conductivity by various modelling techniques uses the properties of the constituent parts, Ref. [82-4], [82-13].

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82.7 CMC: Thermal conductivity

82.7.1 General

The main constituents for CMC composites are shown in Table 82.7.1.

Table 82.7-1 - CMC thermal conductivity: General classification

Composite phase	Conductive	Insulative
Matrix	SiC (CVD) Carbon	Glass-ceramic, e.g. LAS, CAS, MAS.
Fibre reinforcement	Carbon	SiC (Nicalon) Alumina

The conductivity of CMCs is temperature dependent and the temperature range over which they operate can be large. The maximum temperatures quoted are:

- C-C: 2500°C,
- C-SiC: 1900°C,
- SiC-SiC: 1700°C,
- LAS-SiC: 1200°C.

At maximum temperatures, the composites are functioning close to their physical limits and progressive changes in microstructure occur which alters conductivity.

Thermal conductivity of CMC materials is strongly influenced by:

- Fibre type and volume fraction,
- Fibre orientation,
- Matrix microcracking density and porosity.

For bi-directional laminates with C or SiC fibres, the transverse, through-thickness conductivity is appreciably lower than in the fibre directions, Ref. [82-14].

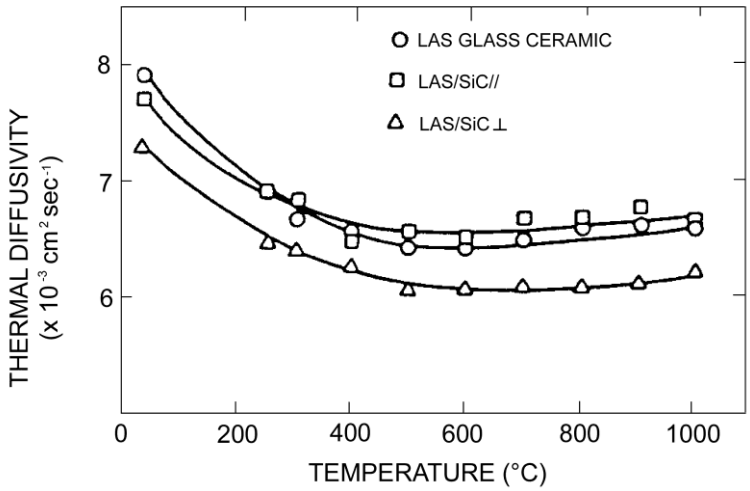
82.7.2 Glass-ceramic matrix composites

The unidirectional thermal conductivity of LAS/Carbon is typically ~2.5W/m/°C.

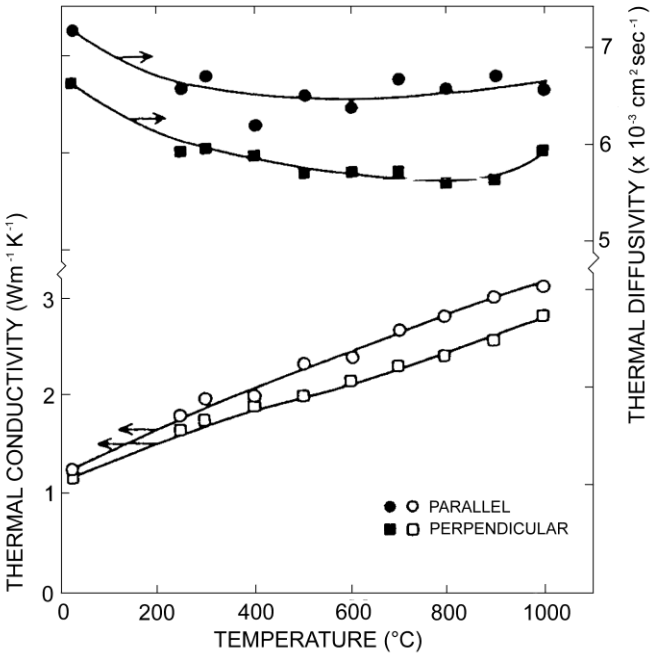
The measured conductivity of amorphous SiC fibres, along with LAS/SiC composites, is shown in Figure 82.7.1, Ref. [82-15].

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A: Experimental data for thermal diffusivity with temperature for LAS matrix with 0, 48 and 49% SiC fibres parallel and perpendicular to the fibre axis.



B: Temperature dependence of thermal conductivity and diffusivity of amorphous SiC fibres, calculated from A.

Figure 82.7-1 - Thermal conductivity and diffusivity for LAS/SiC composites

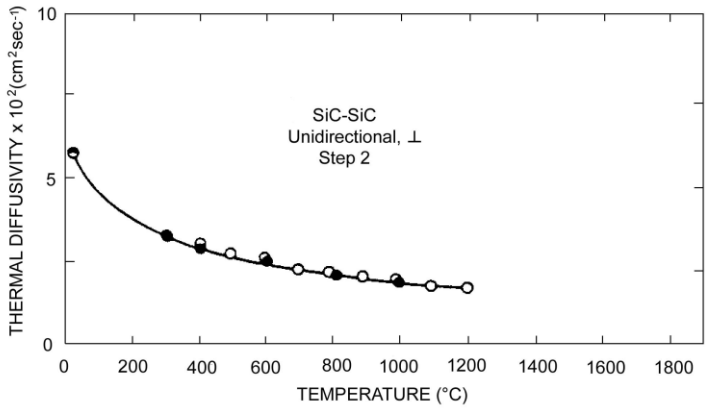
82.7.3 SiC-SiC and C-SiC composites

The properties of some composites are given for, Ref. [82-16]:

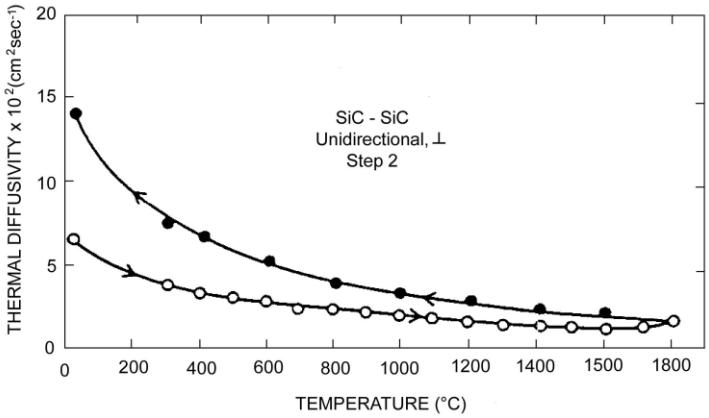
- thermal diffusivity:
  - for SiC-SiC - Figure 82.7.2 (perpendicular to fibres); Figure 82.7.3 (parallel and perpendicular to fibres).
  - C-SiC in Figure 82.7.5, Figure 82.7.6 and Figure 82.7.7
- thermal conductivity: CVD SiC matrix, in Figure 82.7.4.

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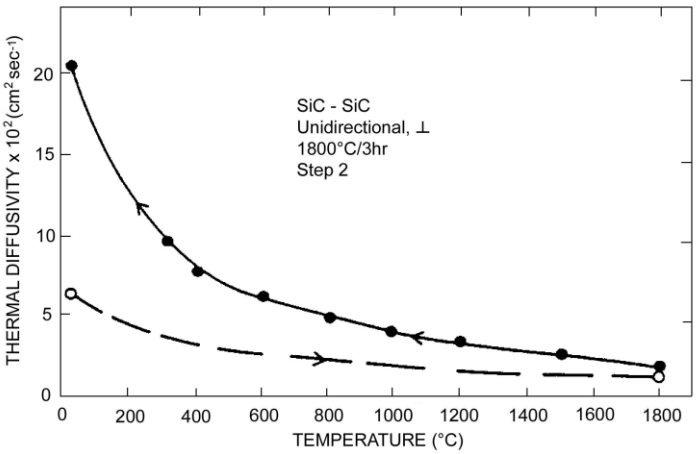
In some cases, the matrix microstructure can change subtly due to heat treatment, which alters the diffusivity, as shown in Figure 82.7.8, Ref. [82-17].



A: Cycled to 1200°C, specimen density: 2.39 g cm^{-3}



B: Cycled to 1800°C, specimen density: 2.47 g cm^{-3}

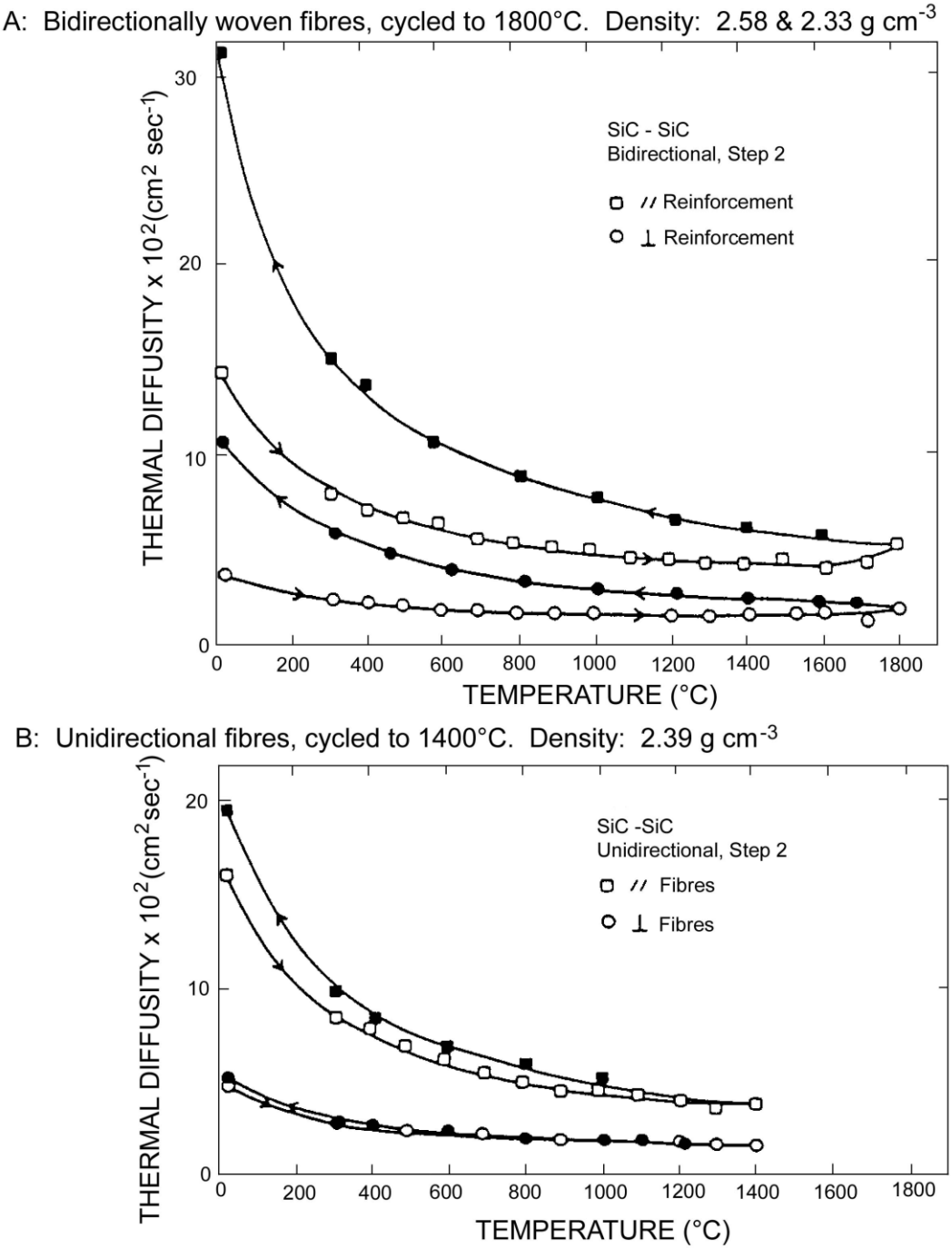


C: Heated rapidly to 1800°C, 3hr hold, cooled at normal rate, specimen density: 2.45 g cm^{-3}

Open symbols = heating; Closed symbols = cooling

Figure 82.7-2 - Thermal diffusivity with temperature for SiC-SiC composite, perpendicular to the fibres

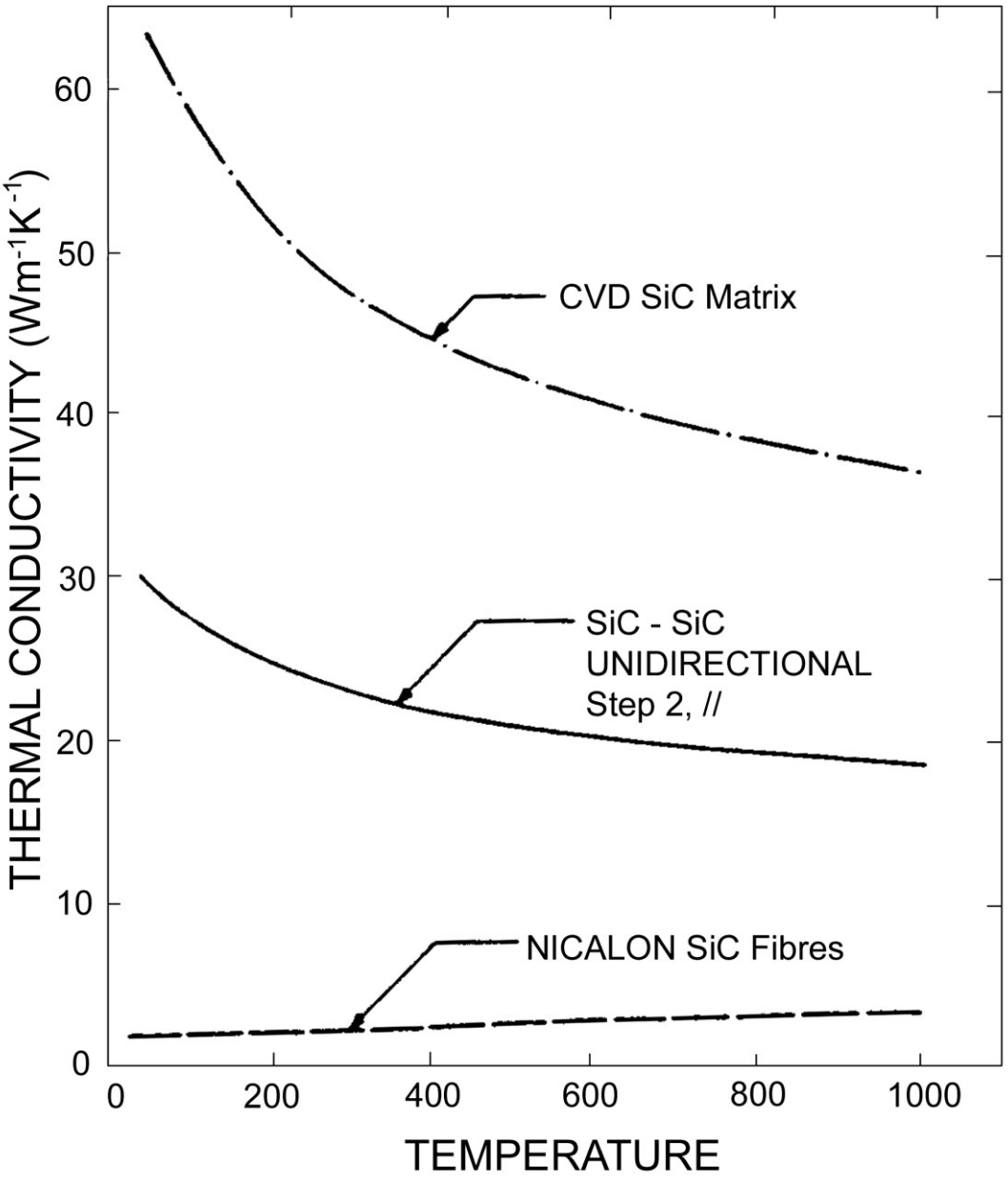
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Open symbols = heating; Closed symbols = cooling

Figure 82.7-3 - Thermal diffusivity with temperature for SiC-SiC composite, parallel and perpendicular to the fibres

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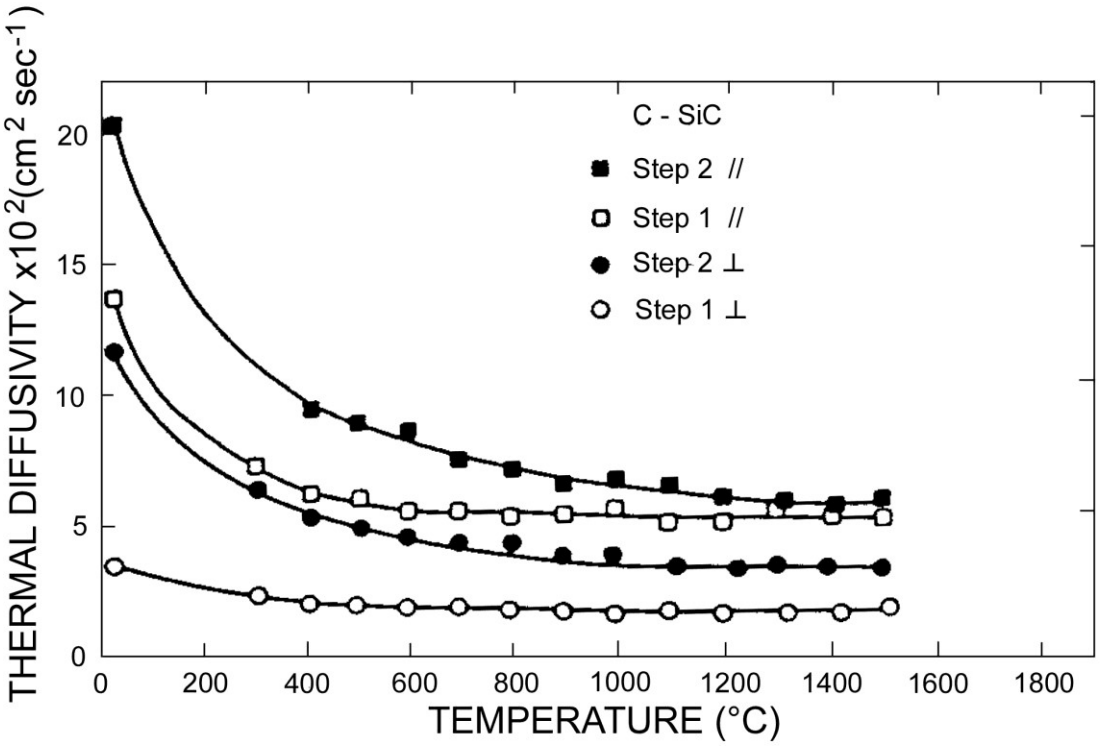


Values determined by composite theory, using the conductivity of Nicalon SiC fibres

Figure 82.7-4 - Thermal conductivity of CVD SiC matrix

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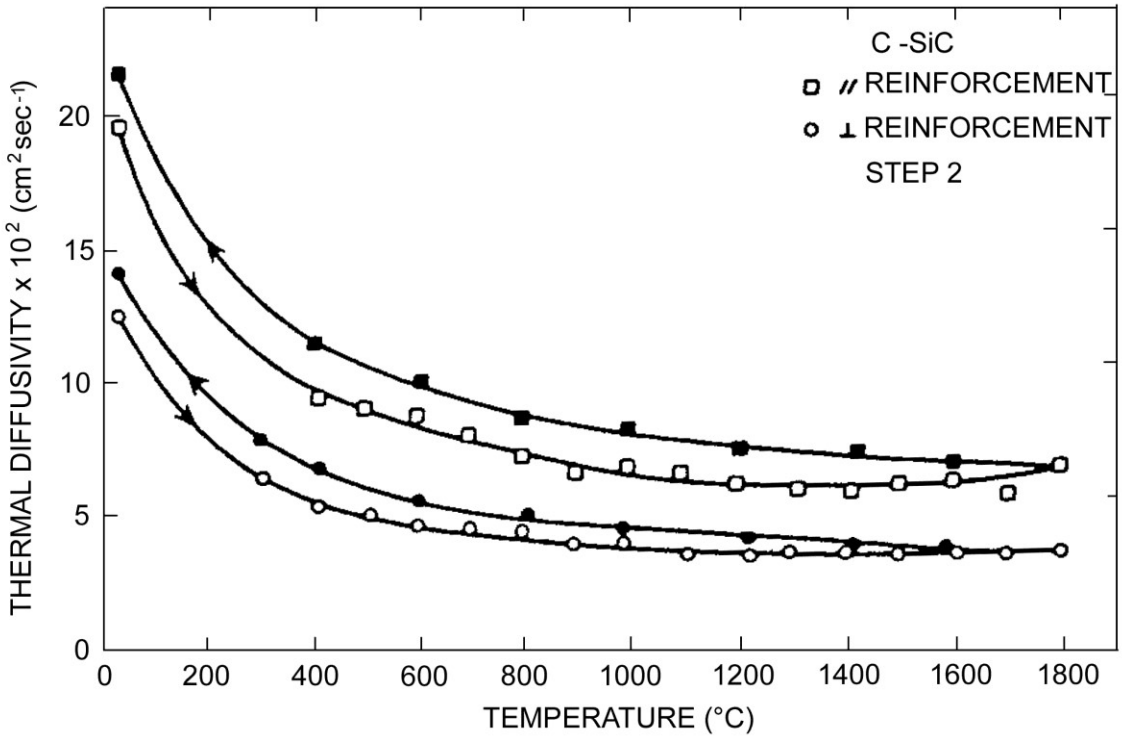
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Key: Values perpendicular and parallel to the fibre.
Step 1: Low density, 1660 and 1540 kg m^{-3}.
Step 2: Intermediate density, 2330 and 2130 kg m^{-3}

Figure 82.7-5 - Thermal diffusivity with temperature to 1500°C for C-SiC composites

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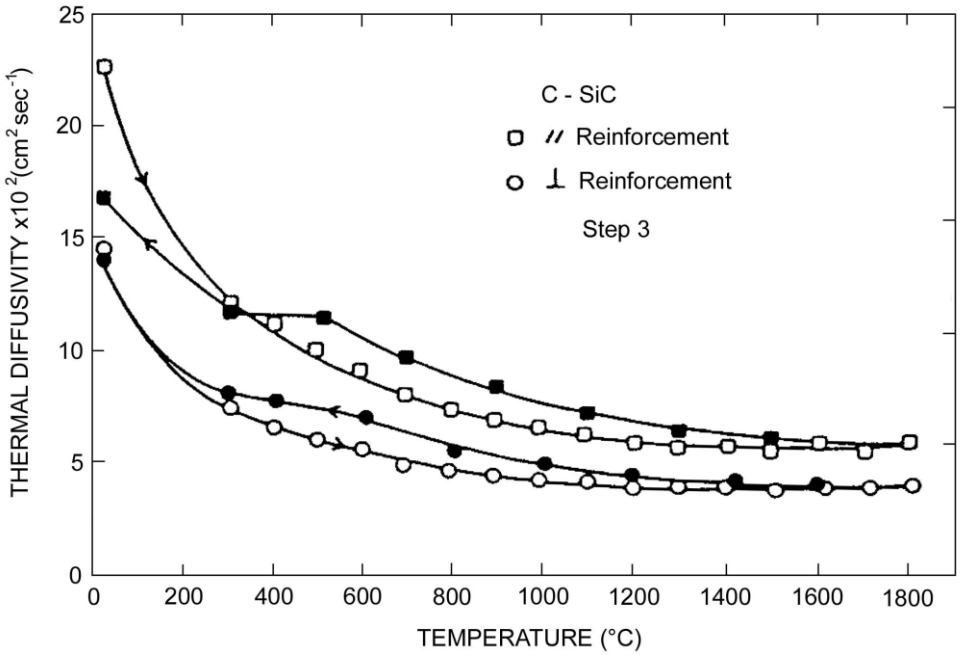


Key: Values parallel and perpendicular to fibre in intermediate density (Step 2) composites, cycled to 1800°C.

Open symbols = heating; Closed symbols = cooling.

Figure 82.7-6 - Thermal diffusivity with temperature for C-SiC composites

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Key: Values parallel and perpendicular to fibre in densified (Step 3) composites, cycled to 1800°C.
Open symbols = heating; Closed symbols = cooling.

Figure 82.7-7 - Thermal diffusivity with temperature for C-SiC composites

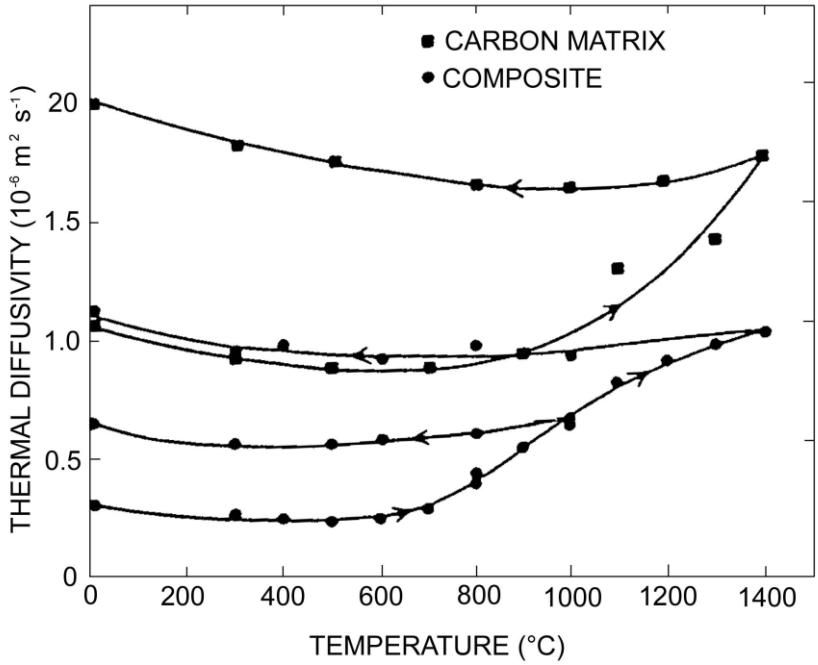


Figure 82.7-8 - Effect of thermal cycling on thermal diffusivity of a carbon matrix and aluminosilicate-carbon composite

The diffusivity of C-C composites is dependent on the overall density, Ref. [82-18]. The diffusivity also falls appreciably as the temperature rises to 2200°C, which is typical of a phonon conductor.

Thermal conductivity for C-C materials can vary between 50W/m/°C and 200W/m/°C, depending on the material characteristics and temperature.

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82.8 Specific heat capacity

Knowledge of material heat capacities is important in determining how much heat energy a structural configuration retains in a high heat flux environment.

Heat capacity, like conductivity, changes as temperatures increase. Specific heat capacity values are often available for individual material elements near room temperature but are less common for MMC and CMC materials.

Figure 82.8.1 shows specific heat values for LAS glass-ceramic and amorphous Nicalon SiC fibres, Ref. [82-15].

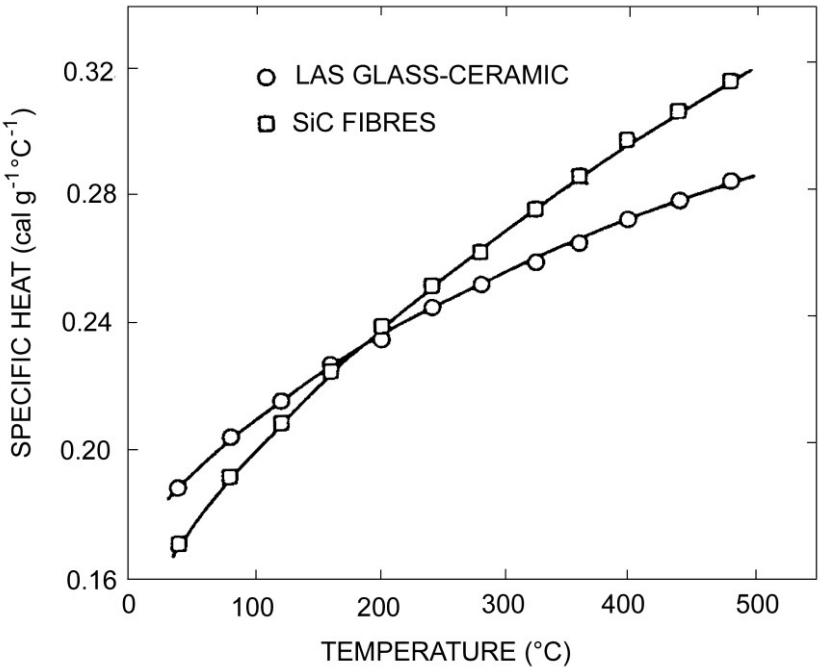


Figure 82.8-1 - Specific heat capacity of LAS glass-ceramic and amorphous SiC fibres

82.9 Surface emissivity

Surface emissivity is an important characteristic for structures experiencing high heat fluxes; such as propulsion units or the leading edges of re-entry vehicles. A high surface emissivity enables heat to be re-radiated, so that heat conducted to underlying structures can be controlled. A desirable emissivity value of 0.85 is often quoted. As an example, Table 82.9.1 shows how the wall temperature of a CMC TPS could vary if the emissivity is low, Ref. [82-19]. This equates to emissions in the 0.4µm to 15µm wavelength band.

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