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Electrical conductivity of silicon carbide composites and fibers

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Abstract

The electrical conductivity, σ , of silicon carbide (SiC) composites and fibers was measured for temperatures between 20 and 1000 °C in order to estimate the magnitude of magnetohydrodynamic effects for liquid metal blankets and a SiC composite structure. Two types of composites were tested: the first type had a matrix made by chemical vapor infiltration (CVI), the second type had a matrix made by polymer-impregnation-pyrolysis (PIP). The electrical conductivity of these materials differed by more than an order of magnitude, 650 (Ω m)⁻¹ for CVI and 22 (Ω m)⁻¹ for PIP composites at 1000 °C. This difference is attributed to the different carbon content of the materials. Several CVI composite samples were irradiated in HFR Petten to doses of 1, 2 and 5 dpa at 750 °C. The thermal conductivity, after 1 dpa, dropped below 30% of the unirradiated value, the drop in electrical conductivity was much smaller. © 2002 Elsevier Science B.V. All rights reserved.

1. Introduction

Silicon carbide (SiC) composites are being developed mainly for structural components in nuclear fusion reactors [1]. More recently, SiC composites have been proposed for non-structural applications, in liquid metal blankets: (a) thermal shielding and (b) electrical insulation, thus, exploiting the low thermal and electrical conductivity of this material [2,3]. Low σ -materials are required in order to minimize magnetohydrodynamic (MHD) effects that result from the interaction of the moving liquid metal and the strong magnetic flux necessary for plasma confinement [4,5]. The electrical conductivity of most SiC composites may differ significantly from that of pure SiC, since carbon/graphite, which is a good electrical conductor, is used as interface material between fibers and matrix.

In order to check, whether or not, the available SiC composites may be used as low σ -materials for liquid metal blankets, the electrical conductivity was measured in the temperature range 20–1000 °C for various types of

SiC composites and fibers: CG Nicalon, Hi Nicalon, Hi Nicalon Type S and Tyranno SA. The composites tested were made with CG Nicalon or Hi Nicalon fibers, the matrix being produced by chemical vapor infiltration (CVI) or polymer-impregnation-pyrolysis (PIP).

2. Materials and experimental procedure

Table 1 gives a listing of the SiC composite materials used for the tests. The Cerasep® 2N-1 produced by Societe Europeenne de Propulsion (SEP) has a two dimensional (2D) structure of CG Nicalon NL207 fibers and a matrix produced by CVI with a carbon interfacial layer of unknown thickness between fibers and matrix. The Cerasep[®] 3N-1 has a multilayer 3D texture called GUIPEX[®] [6]. The third texture direction is created by linking the layers during weaving, so the risk of delamination is reduced when shaping and densifying the parts [3]. Both materials, Cerasep® 2N-1 and Cerasep® 3N-1, had equal components, CG Nicalon fibers and a CVI matrix. Therefore, their physical properties should be similar. The third composite material had a CVI matrix and Hi Nicalon fibers instead of CG Nicalon. The fourth sample was part of an experimental batch developed by ENEA, Italy, and FN Spa, Italy, using PIP.

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Table 1SiC composites used for the tests

Company	Trade name	Fiber	Matrix
SEP, France SEP SEP ENEA/FN, Italy	Cerasep [®] 2N-1 Cerasep [®] 3N-1 –	CG Nicalon [®] CG Nicalon [®] Hi Nicalon [®] CG Nicalon [®]	CVI CVI CVI PIP

It had a 2D structure of CG Nicalon fibers and no interfacial layer between fibers and matrix. All specimens had a rectangular cross section Q of typically 2 mm height, 8 mm width and 70 mm length. Both ends of the specimens were connected to a constant current source, which provided a current of magnitude *I*. The potential drop V was measured over the distance L with the aid of two knife-edge ridges pressed on the sample. The conductivity $\sigma = (I/V)(L/Q)$ was calculated by taking the mean value of two voltage and current measurements for normal and reversed current direction in order to minimize the effect of contact voltages. The samples were placed into an oven under helium atmosphere. The oven temperature, measured by two type K thermocouples, was increased or decreased at a rate of about 1-2°/min up to 1000 °C and back to room temperature.

The fibers tested are listed in Table 2. For the resistance measurements, a bundle of the fibers in the asreceived condition was mounted into the holder.

3. Results

3.1. Conductivity of fibers

Fig. 1 shows the electrical conductivity, σ , of the three Nicalon fibers as a function of temperature in the range 20–1000 °C. Solid symbols stand for heating, open symbols for cooling conditions. At room temperature, CG Nicalon fibers had the highest conductivity: 120 $(\Omega m)^{-1}$ in comparison to 30 $(\Omega m)^{-1}$ for Hi Nicalon and 0.3 $(\Omega m)^{-1}$ for Hi Nicalon Type S fibers. For the Hi Nicalon and Hi Nicalon Type S fibers, the conductivity increased monotonically with temperature in the whole interval 20–1000 °C. The cooling curves lay slightly below the heating curves. The situation was different for

400 Hi Nicalon conductivity [Ω m]⁻¹ 300 CG Nicalon 200 100 Туре S 0 1000 C 200 400 600 800 1200 temperature [°C]

Fig. 1. Electrical conductivity of Nicalon type fibers. Solid symbols stand for heating, open symbols for cooling conditions.

CG Nicalon fibers. There was an increase in conductivity from 20 °C to about 890 °C. A further increase in temperature resulted in a drop in conductivity, therefore, the cooling $\sigma(T)$ -curve stayed significantly below the heating curve. The decrease in σ at temperatures > 900 °C may have the same cause as the known thermal instability of CG Nicalon fibers observed for temperatures above about 930 °C [7]. However, for the composites, no such drop in σ was observed in the temperature range 20–1000 °C. This may be explained by assuming that the contribution of the fibers to the total conductivity of the composite is small in comparison to the contribution of the matrix and the carbon interfacial layer between fibers and matrix. Carbon is an excellent electrical conductor.

In Fig. 2, σ of Tyranno SA fibers is plotted as a function of temperature. σ increased from RT to about 500 °C, as expected, but then the conductivity dropped with *T*, followed by an increase up to 1000 °C. An energy dispersive spectroscopy-analysis of Tyranno SA fibers conducted before and after the $\sigma(T)$ -measurements suggested that the fiber were covered by carbon, which at a temperature of 500 °C was removed by oxidation, thus lowering σ . The drop in σ was a first run effect, since the $\sigma(T)$ -values measured in a second and third run were close to the those of the cooling curve shown in Fig. 2. So, the initially high electrical

Table 2 SiC fibers used for the tests

Company	Trade name	Diameter (µm)	C/Si ratio	Oxygen (wt%)
Nippon Carbon, Japan	CG Nicalon	14	1.31	11.7
Nippon Carbon	Hi Nicalon	14	1.39	0.5
Nippon Carbon	Hi Nicalon Type S	12	1.05	0.2
UBE, Japan	Tyranno SA	7.5	1.0	_



Fig. 2. Electrical conductivity of Tyranno SA fibers. Solid symbols stand for heating, open symbols for cooling conditions.

conductivity of the Tyranno SA fibers tested may be attributed to a surface carbon layer.

3.2. Conductivity of composites

The three composites, 2D and 3D SEP, that are made by applying the same production procedure, carbon coated fibers + CVI matrix, have a similar $\sigma(T)$ -function between 50 and 1000 °C, (see Fig. 3). σ increased from 200 (Ω m)⁻¹ at room temperature to about 650 (Ω m)⁻¹ at 1000 °C. The slight difference in $\sigma(T)$ -function of the Hi Nicalon material and its higher temperature coefficient may be attributed to the different fiber type used. The $\sigma(T)$ function of the two fibers, Hi Nicalon instead of CG Nicalon, had the same characteristics as the respective composites (see Fig. 1). In comparison, the electrical conductivity of the fourth composite made by ENEA/FN is more than an order of magnitude lower. Its matrix is produced by PIP and therefore, its structure will differ from that of the CVI matrices, but, more



Fig. 3. Electrical conductivity of composites.

important for the electrical conductivity, the material had no carbon layer between fiber and matrix. However, the resulting magnitude of $\sigma \sim 20 \ (\Omega m)^{-1}$ at 1000 °C is still higher than the limiting value of $\sim 2 \ (\Omega m)^{-1}$, which was postulated in recent design studies for a suppression of MHD effects [5]. Figs. 1-3 suggest that the total conductance of a composite, S, may be expressed as the sum of the conductances of its components just as it is the case for a simple network: $S = \sum \sigma_i Q_i / L$, where σ_i is the conductivity of the *i*th component, Q_i is its cross section and L the specimen length. According to this relationship, the conductivity of the composites may be calculated. Obviously, a carbon interfacial layer between fiber and matrix, even if its thickness lies in the order of 0.1 µm may give the main contribution to the overall conductivity of the composite, since the σ of carbon is about two orders of magnitude higher than that of SiC. For this reason, a significant reduction in σ may be obtained by using less conductive materials for the interfacial layer between fibers and matrix of the composite.

3.3. The effect of neutron irradiation

Six Cerasep 2N-1 samples were irradiated in the HFR Petten to doses of 1, 2 and 5 dpa at a temperature of 750 °C. Thereafter, the thermal and electrical conductivity was measured at 20 °C. The results are reported in Fig. 4. The normalized conductivities are plotted versus dose. Fig. 4 confirms the well-known result that thermal conductivity, after irradiation at 750 °C already after doses in the order of 1 dpa, is reduced to about one third of the pre-irradiation value. The drop in electrical conductivity, σ , is much smaller and seems to be a linear function of dose for the Cerasep samples tested. For this material, the carbon interface will con-



Fig. 4. The normalized electrical and thermal conductivity is plotted versus the irradiation dose $(1dpa = 10^{25} n m^{-2})$. The samples were irradiated in HFR Petten.

tribute significantly to the total electrical conductivity of the composite. It can be speculated that this interface is affected only slightly by the irradiation. The drop in σ may be attributed mainly to the irradiation induced structural damage of the samples: the CG Nicalon fibers are known to shrink during irradiation [8]. This shrinkage leads to a separation of the fibers from the matrix and, thus, to a reduced electrical conductivity.

4. Conclusions

The electrical conductivity of SiC fibers and SiC composites was measured for the temperature range 20–1000 °C in order to estimate the MHD induced pressure drop for a liquid metal blanket and a SiC composite structure. The fibers tested were CG Nicalon, Hi Nicalon, Hi Nicalon Type S and Tyranno SA, the composites were made with CG Nicalon or Hi Nicalon fibers, and their matrices were produced by CVI or PIP. The tests show that:

- The electrical conductivity, σ , of the materials, except that of CG Nicalon and Tyranno SA fibers, increased monotonically with temperature in the range 20–1000 °C.
- The electrical conductivity of a SiC composite may be reduced significantly by avoiding high conductiv-

ity materials, such as carbon, for the interfacial layer between fibers and matrix.

• Neutron irradiation of 5 dpa reduced by about 20% the electrical conductivity of a CVI composite with CG Nicalon fibers.

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