THE KFIB EXPERIMENT - G. E. Youngblood, D J. Senor and R. H. Jones (Pacific Northwest National Laboratory*), W. Kowbel (MER Corporation), and A. Kohyama (Kyoto University)

OBJECTIVE

The primary objective of the experiment called "KFIB" is to assess the thermal conduction **(K)** properties of several advanced SiC **FIB**ers before and after irradiation. A secondary goal is to assess the thermal conductivity of SiC/SiC composites made from these fibers (with various SiC-type matrices and architectures) before and after irradiation and to model the transverse and in-plane thermal conductivity of these composites as a function of temperature and dose.

SUMMARY

Several rod-shaped specimens with uniaxially packed fibers (Hi-Nicalon™, Hi-Nicalon™ Type S, Tyranno™ SA and Amoco K1100™ types) in a pre-ceramic polymer matrix were fabricated. By using appropriate analytic models, the bare fiber thermal conductivity (K_f) will be determined as a function of temperature up to 1000°C before and after irradiation for samples cut from these rods. Preliminary thermal conductivity data for unirradiated fibers (Hi-Nicalon™ and Tyranno™ SA-B SiC and K1100™ graphite) and for three types of unirradiated composites made from these fibers (2D-Nicalon S/SiC multilayer/CVI-SiC, 3D-Nicalon S/PIP-SiC, and 2D-8HS Tyrannohex™ HP) are presented.

PROGRESS AND STATUS

The KFIB experiment is coordinated at the Pacific Northwest National Laboratory (PNNL), but involves several other organizations. Other organizations have furnished materials, materials analysis or irradiation facilities for the experiment. The cooperating organizations and points of contact are:

Pacific Northwest National Laboratory (PNNL) in Richland, WA; contact Jerry Youngblood, (ge.youngblood@pnl.gov)
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SiC Fibers

The thermal conductivity of the following advanced SiC and C fibers will be assessed before and after irradiation: Hi-Nicalon™, Hi-Nicalon™ Type S, Tyranno™ SA, and Amoco K1100™ as described previously [1]. These fibers will be used to make SiC/SiC (or C/SiC) composites with

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uniaxial alignment of the fibers. The composites will be fabricated into rod shapes that conveniently can be sliced into several discs for measurement of the thermal diffusivity along the fiber axial direction. The matrix component will purposely be made amorphous from pre-ceramic polymers (Ceraset™ or KION™) so that the fiber contribution to the overall composite K-value will be dominated by the fiber contribution. To further enhance the accuracy of the fiber K-value determinations, the composite rods will contain a relatively high (>60%) fiber packing fraction.

SiC/SiC Composites and Monolithic CVD-SiC

Several types of SiC/SiC composites made with the advanced fibers and with different architectures will be included in the experimental matrix. Samples of Morton high-purity CVD-SiC will also be included in each capsule to serve as passive temperature and radiation damage monitors [2].

Irradiation Tests and Schedule

Two irradiation tests are planned. A low temperature irradiation test will take place in the ATR reactor at 300°C to a dose of 3.6 dpa-SiC. A moderate and a high temperature irradiation test will take place in the HFR Petten reactor at 627°C and 1027°C to a dose of about 2.5 dpa-SiC. The ATR irradiation will commence in November, 2000 and will be completed by May, 2001; the HFR Petten irradiation will commence in January, 2001 and will be completed by July, 2001. The sample test matrix is given in Table 1.

Table 1. KFIB sample test matrix

Fiber Discs (6.2 mm dia x 2.0 mm thk)	**HFR Petten**	**ATR**
Hi-Nicalon (parallel)	4	2
Hi-Nicalon (perpendicular)	2	2
Tyranno SA-B (parallel)	4	2
Nicalon Type S (parallel)	6	2
Amoco K1100 graphite (parallel)	0	2
Composite Discs (6.2 mm dia x 2.0 mm thk)		
2D-Nicalon S/SiC multilayer/CVI-SiC	6	4
3D-Nicalon S/PIP (Ceraset 1400C)	4	4
2D-8HS Tyrannohex HP (100% transverse)	4	3
2D-8HS Tyrannohex HP (50% transverse)	0	2
2D-5HS Nicalon S/CVI-SiC w K1100(z-stitch)	2	0
Morton High-Purity CVD-SiC Reference		
CVD-SiC (6.0 mm dia x 2 mm thk discs)	3	3
CVD-SiC (25.5 mm long x 1.85 mm sq bars)	6	0

Ten fiber, thirteen composites made with these fibers and three CVD-SiC thermal diffusivity disc samples have been inserted into the ATR reactor. Sixteen fiber and sixteen composite disc samples made with these fibers and nine CVD-SiC reference samples will be irradiated in the HFR Petten reactor.

The Hi-Nicalon™ and Hi-Nicalon™ Type S SiC fibers were fabricated by the Nippon Carbon Co. (Yokohama, Japan) using an electron-beam radiation curing process. The Tyranno™ SA-B fiber was fabricated by Ube Industries Ltd. (Ube City, Japan) using an oxidative curing process and high temperature (1800°C) sintering. It is noted that the tested Tyranno™ SA-B fiber, a new smaller diameter version of Tyranno™ SA, has improved thermal creep and high temperature strength compared to the Tyanno™ SA fiber version [3].

The 2D-Nicalon™ S/SiC multilayer/CVI-SiC composite was made by Hypertherm High Temperature Composites, Inc. (Huntington Beach, CA) by the same procedure as a similar composite examined by Snead, et al [4], except the Hi-Nicalon™ fiber was replaced with Hi-Nicalon™ type S fiber. Snead reported that the Hi-Nicalon™ multilayer composite, where the fibers were coated by CVD with four alternate layers of C/SiC prior to matrix infiltration, exhibited the least strength degradation after low temperature neutron irradiation compared to composites with either a single layer PyC or a "porous SiC" fiber coating. Replacing the Hi-Nicalon™ fiber with the more radiation resistant Hi-Nicalon™ type S fiber may improve the radiation strength stability of the multilayer composite even further. However, thermal conductivity results have not been reported for the Hi-Nicalon™ multilayer composite until now.

The fabrication and mechanical properties of the 3D-Nicalon™ S/PIP-SiC composite, contributed by JAERI, were reported earlier by Yamada, et al [5]. The 3D fiber architecture for this composite had an X/Y/Z pattern with either 1/1/0.1 or 1/1/0.2 relative fiber volume ratios. The thermal conductivity was determined in the Z-direction.

The fabrication and characteristics of the 2D-8HS Tyrannohex™ HP composite are fully described in an accompanying paper in this volume [6].

A 2D composite with Hi-Nicalon™ type S SiC fiber in the X-Y plane but studded with K1100™ graphite fiber rods in the Z-direction is the final composite included in the KFIB test matrix. This composite, fabricated by MER Corp. using a special PIP matrix and a high temperature treatment, is an attempt to improve the transverse thermal conductivity by inserting highly conductive K1100™ fibers in the Z-direction [7].

The thermal diffusivity and conductivity for a representative number of disc-shaped composite samples were determined by the laser flash method described in detail previously [8].

Results

During the latest period, data for the unirradiated thermal conductivity of Tyranno™ SA-B fiber were obtained and added to the previously reported fiber conductivity data for unirradiated Hi-Nicalon™ SiC fiber and K1100™ graphite fiber as shown in Fig. 1 [1]. The thermal conductivity for the unirradiated Tyranno™ SA-B SiC fibers ranges from about 45 W/mK at ambient down to 18 W/mK at 1000°C and are considerably higher than similar values for the Hi-Nicalon™ fiber. The thermal conductivity of the Tyranno™ SA fiber version, reported to be 65 W/mK at ambient, is higher still [9].

The transverse thermal conductivity results for the 2D-Nicalon™ S/SiC multilayer/CVI-SiC and the 3D-Nicalon™ S/PIP-SiC materials are presented in Figs. 2(a-b), respectively.

In Fig. 2(a), the thermal conductivity values for different samples of the 2D multilayer composite (CVI-SiC matrix) are distributed over a wide range between 5-14 W/mK. In general, samples with higher bulk density values exhibited the higher thermal conductivity values. The sample densities, which are used as labels to identify corresponding data in the figure, ranged from a high value of 2.59 g/cc down to 2.31 g/cc. Usually high quality SiC/SiC made with a CVI-SiC matrix exhibits a bulk density of 2.5 g/cc or better. The thermal conductivity of such material when made with Nicalon CG fiber is about 10 WmK at ambient, and when made with Hi-Nicalon it is about 13 W/mK [8]. Only for one multilayer sample did the density exceed 2.5 g/cc, and for that sample the thermal conductivity at ambient was slightly more than 14 W/mK. All samples with densities less than 2.5 g/cc exhibited thermal conductivity values less than 10 W/mK at ambient. These results suggest that achieving high quality composite by the CVI-method might be more difficult when multilayer coatings are used. Perhaps when using multilayer coatings there is a greater tendency to seal off the fiber bundles and prevent adequate infiltration. Even when the infiltration was adequate, the thermal conductivity values for this type of 2D SiC/SiC composite were significantly below desired values for the fusion application [10].

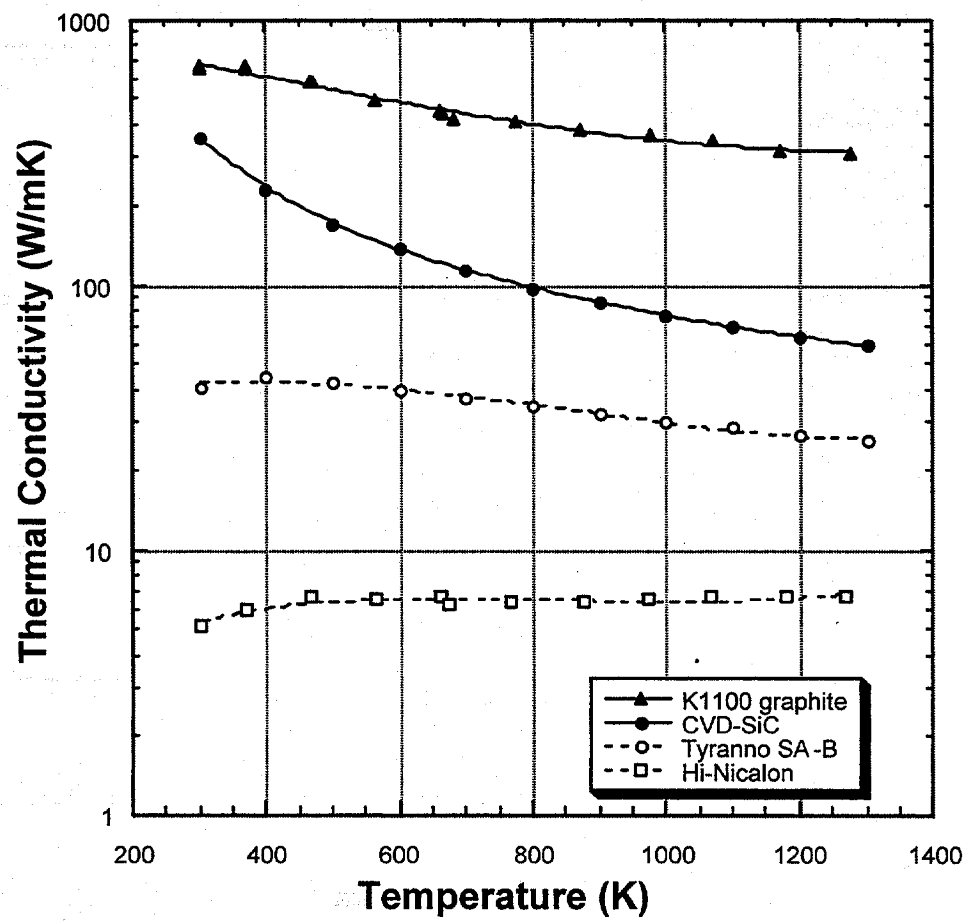

Figure 1. The measured thermal conductivity values for K1100™ graphite, Tyranno™ SA-B and Hi-Nicalon™ fibers compared to the thermal conductivity of high-purity, monolithic CVD-SiC.

In Fig. 2(b), the average thermal conductivity values for the 3D-Nicalon S composite (PIP-SiC matrix) are plotted as a function of temperature. The values all fall within a narrow range of about 5-6 W/mK with little temperature dependence indicated. The thermal conductivity values for the composite made with 0.2 relative fiber volume ratio in the Z-direction were only slightly larger than values for composite with a 0.1 volume ratio. The conductivity values representing materials with each fiber volume ratio (0.2 or 0.1) were determined by averaging values made for three different samples selected from each group. The resulting values agree fairly well with values measured by Yamada for similar materials, except Yamada's values exhibited a slightly larger spread between composites with different fiber volume ratios [5]. As expected, Yamada observed higher thermal conductivity values (by ≈20%) for the Z = 0.2 group which contained approximately twice as many Nicalon S fibers aligned in the heat flow direction as the Z = 0.1 group. The bulk density values for our Z = 0.1 and 0.2 groups were 2.40 and 2.24 g/cc, respectively. The lower average density observed for our Z = 0.2 group likely counteracted the benefit of having additional fibers aligned in the Z-direction; so only slight differences in thermal conductivity values were observed between the two groups.

In general, the observed thermal conductivity values for these 3D-composites with a PIP-SiC matrix were considerably less than values observed for 2D-SiC/SiC composites made with a CVI-SiC matrix. Apparently, the low thermal conductivity of the continuous PIP-SiC matrix phase dominates the conduction process in these materials, and any benefits derived from the Z-stitching were minimal. These results suggest that a PIP-SiC matrix must be annealed at high enough temperature to completely crystallize the matrix before the thermal conductivity will be significantly improved. At the same time the formation of shrinkage cracks in the matrix must also be avoided (highly unlikely). Only then is there a possibility for attaining fusion thermal conductivity goals for SiC/SiC composites made with a PIP-type matrix.

The results for the 2D-8HS Tyrannohex HP are presented in a separate paper in this volume [6]. The thermal conductivity results for the 2D-5HS Nicalon S/CVI-SiC w K1100 (z-rods) will be reported later.

FUTURE WORK

The thermal conductivity for advanced SiC fibers (Hi-Nicalon™, Hi-Nicalon™ Type S and Tyranno™ SA-B) and for graphite fibers (K1100™) will be analyzed before and after irradiation. Similar analysis will be performed for composites made with these fibers by using appropriate thermal conductivity models. The results should be available by the end of 2001.

ACKNOWLEDGEMENTS

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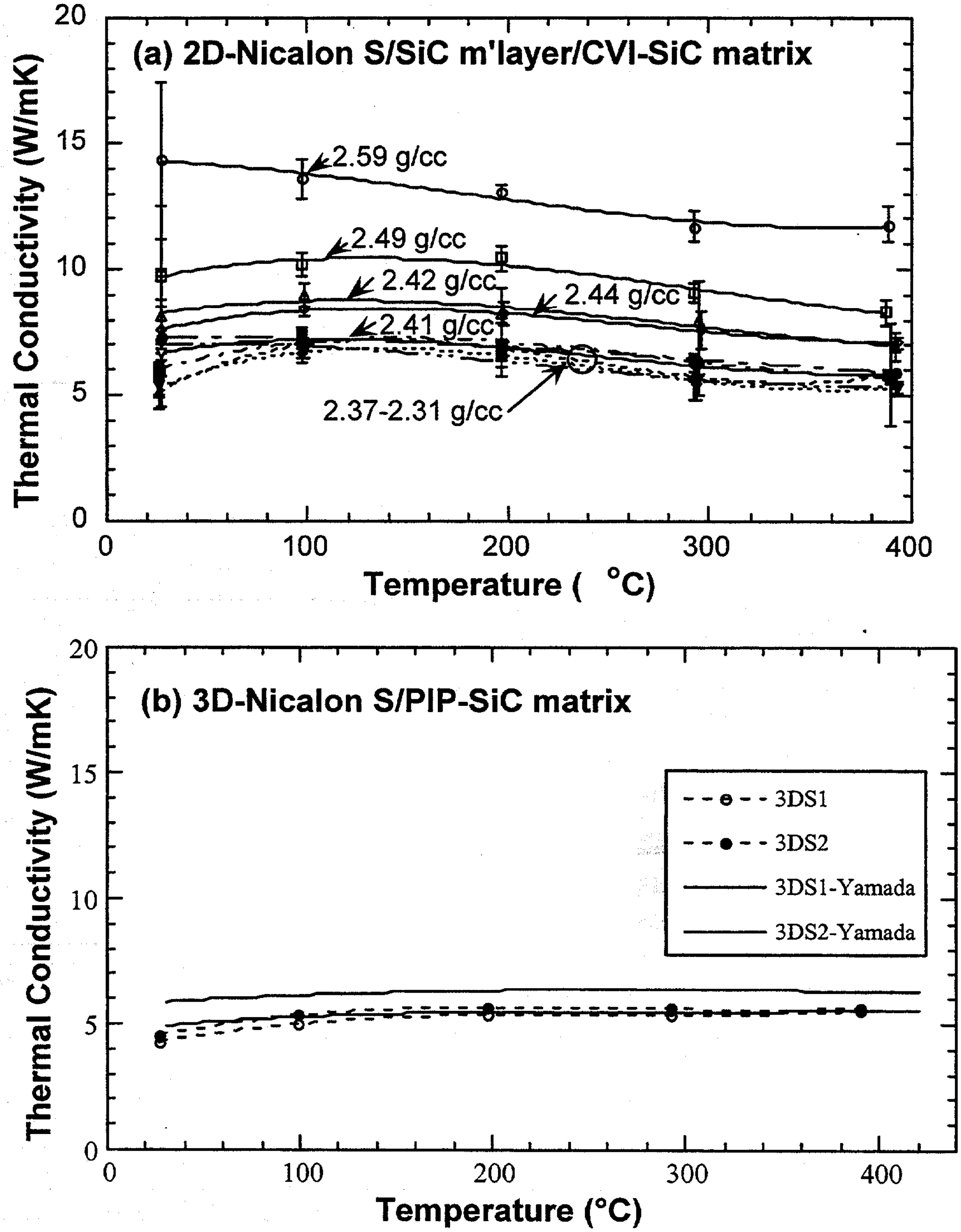

Figures 2(a-b). Thermal conductivity of (a) 2D-Nicalon S/SiC multilayer/CVI-SiC and (b) 3D-Nicalon S/PIP-SiC. In Fig. 2(b), the X-Y-Z fiber volume ratios were 1-1-0.1 and 1-1-0.2 for the samples labeled 3DS1 and 3DS2, respectively.

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