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Effects of contact resistance on electrical conductivity measurements of SiC-based materials

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ABSTRACT

A combination 2/4-probe method was used to measure electrical resistances across a pure, monolithic CVD-SiC disc sample with contact resistance at the SiC/metallic electrode interfaces. By comparison of the almost simultaneous 2/4-probe measurements, the specific contact resistance (R_c) and its temperature dependence were determined for two types (sputtered gold and porous nickel) electrodes from room temperature (RT) to ~973 K. The R_c-values behaved similarly for each type of metallic electrode: $R_c > \sim 1000\ \Omega\,\text{cm}^2$ at RT, decreasing continuously to ~1–10 $\Omega\,\text{cm}^2$ at 973 K. The temperature dependence of the inverse R_c indicated thermally activated electrical conduction across the SiC/metallic interface with an apparent activation energy of ~0.3 eV. For the flow channel insert application in a fusion reactor blanket, contact resistance potentially could reduce the transverse electrical conductivity by about 50%.

1. Introduction

Recently, we have reported on measurements of the transverse electrical conductivity (EC) of a silicon carbide fiber reinforced (SiC_f) silicon carbide composite made by chemical vapor infiltration (CVI), referred to as SiC_f/CVI-SiC, a potential flow channel insert (FCI) material [1]. When using a simple 2-probe dc method to measure the effective EC of SiC_f/CVI-SiC with a thin chemical vapor deposition (CVD) SiC seal coat as a function of temperature up to 1073 K, we encountered an unexpectedly high overall electrical resistance. The high resistance values were attributed to a large contact resistance contribution at a solid metal electrode-CVD SiC seal coat interface, as also observed by Morley et al. [2]. Morley's experiment, carried out at 573–823 K with a solid CVD-SiC disc exposed to liquid Pb–Li metal alloy electrodes, simulated somewhat the operating temperature and chemical conditions expected in the operation of an FCI [3–5]. Because one purpose of an FCI is to reduce MHD-generated transverse electrical current, an additional contact resistance at the SiC–liquid metal interface would be beneficial. Therefore, understanding the behavior of contact resistance at such an interface is important in the design and operation of an FCI.

In this work, we used a combination 2-probe/4-probe setup to simultaneously measure the electrical resistance across a pure, monolithic CVD-SiC disc sample as a function of temperature. The true resistance (or the EC) of the CVD-SiC material was determined by the 4-probe method without interference of contact resistance; while the larger resistance determined simultaneously by the 2-probe method contained the contact resistance contribution in series with the sample resistance. By simple comparison of the simultaneous 4-probe/2-probe resistance values at each temperature, the specific contact resistance (R_c) and its temperature dependence were determined.

2. Material and methods

A 5-mm thick plate of high-purity, monolithic CVD-SiC was obtained from Rohm and Haas (Woburn, MA). All resistance or EC determinations were made for a single CVD-SiC sample disc (1-cm dia. × 5-mm thick) cut from this plate. Two centrally located holes (1-mm dia. × 1-mm deep) were bored into opposite disc faces in which to seat 4-probe potential leads electrically isolated from common 2-probe current and potential leads connected to the disc faces. The disc sample was polished down to 0.5 μm grit, degreased, and carefully cleaned with a mild HF solution before applying metal electrodes, either sputtered gold or porous nickel electrodes.

Initially, gold electrodes were applied to the disc faces using a Ladd 30800 sputter coater. The disc sample was then mounted in an enclosed alumina tube fixture in which a low-flow reducing atmosphere (3% H_2 in argon) was maintained. For the 2-probe measurements, gold current and voltage wires were joined to gold foil that was pressed onto disc faces with sputtered gold electrodes by external spring-loaded alumina pushrods. For the 4-probe potential measurements, platinum wires with beaded ends were threaded inside alumina thermocouple tubes that were independently spring-loaded and pressed into the centrally located holes in the disc faces. Separately spring-loading the pushrods and the

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central potential probes was essential to compensate for thermal expansion of the components and to provide a steady pressure on the electrodes during temperature cycling. Cyclic I–V voltammograms (DC current vs. voltage) were performed from ±0.1 V at a scan rate of 5 mV/s with a Solartron™ 1285 potentiostat. A National Instruments™ NI USB-9481 electromechanical relay was utilized to automate the almost simultaneous 2/4-probe measurements. Labview™ software was used to control the alumina tube furnace temperature, potentiostat and relay, and store the data. Over the tested voltage range all the I–V data were linear, which indicates "Ohmic-type" behavior. Electrical resistance values were calculated from the slopes of the cyclic voltammograms; EC-values were calculated from the appropriate sample geometry assuming linear current patterns. A schematic view of the instrumental setup is given in Fig. 1; a photograph of the alumina tube fixture and the 1.0-cm. diameter CVD-SiC disc sample prior to application of the Au or porous Ni electrodes is shown in Fig. 2.

Measurements were made in 40° steps during three different temperature cycles, each cycle attaining a successively higher maximum temperature: RT to 673 K then back to RT, then RT to 823 K and back to RT, and finally RT to 973 K and back to RT. Each temperature cycle run required about 20 h. At each step the temperature was measured and then remeasured 10 min later. If the two temperature measurements were within 2 K it was assumed that temperature equilibrium had been attained and I–V measurements proceeded. A 2-point voltammogram was performed first, and then a 4-point measurement immediately followed. When finished the temperature was changed to the next temperature step at a rate of 5 K/min. After I–V measurements for the three temperature cycles with Au electrodes were completed, the sample was removed from the fixture, and the sputtered Au electrodes were removed by light repolishing. The now bare disc sample was retreated with the HF solution; and then porous nickel electrodes were applied. To do this, Baker™ nickel oxide (NiO) was made into a paste using ESL-450 binder. The sample was screen printed with a 4-mil print on both surfaces. The sample was remounted in the fixture with gold contacts now attached to a nickel foil pressed by the alumina pushrods onto the NiO-coated SiC disc surfaces. The sample was heated to 673 K in 3% H_2 in argon and given time to reduce the NiO in situ to porous Ni and cooled to RT. Then the previous temperature cycles and I–V measuring procedures were repeated for the porous Ni electrode case.

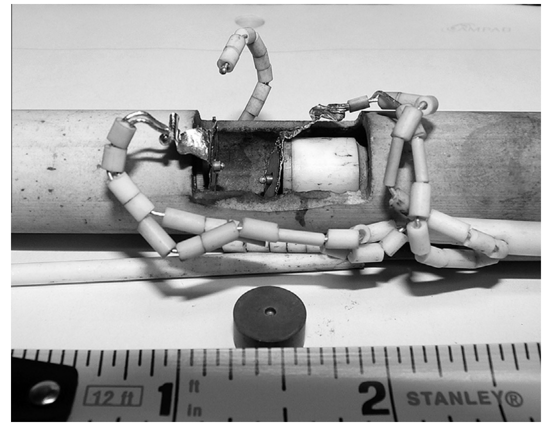

Fig. 2. Alumina tube and sample mounting fixture with the 1.0-cm diameter CVD-SiC disc sample removed and shown below. Two pairs of I–V leads for 2-probe measurements are soldered to gold or nickel foil electrodes that press onto the sample disc with deposited sputtered Au or porous Ni electrodes, respectively. The gold or nickel foils now act only as current leads for 4-probe measurements. The 4-probe potential leads (Pt wire with beaded end held inside an electrically insulating alumina thermocouple tube) extend through small holes cut into the foil current connections on each side of the disc sample and are seated into 1.0-mm deep centrally located holes in the sample.

3. Results and discussion

The measured 2/4-probe transverse resistance values for a monolithic CVD-SiC disc are presented in Fig. 3. For clarity, only the data for the third temperature heating leg to 973 K are shown. In Fig. 3, the measured transverse resistance 2-probe and 4-probe resistance values are shown. They all decrease considerably with increasing temperature. The 2-probe values exceed the 4-probe values for both the porous Ni and sputtered Au electrodes; the differences represent the contact resistances between SiC and the metal electrodes for each case. Note that the 4-probe measurements

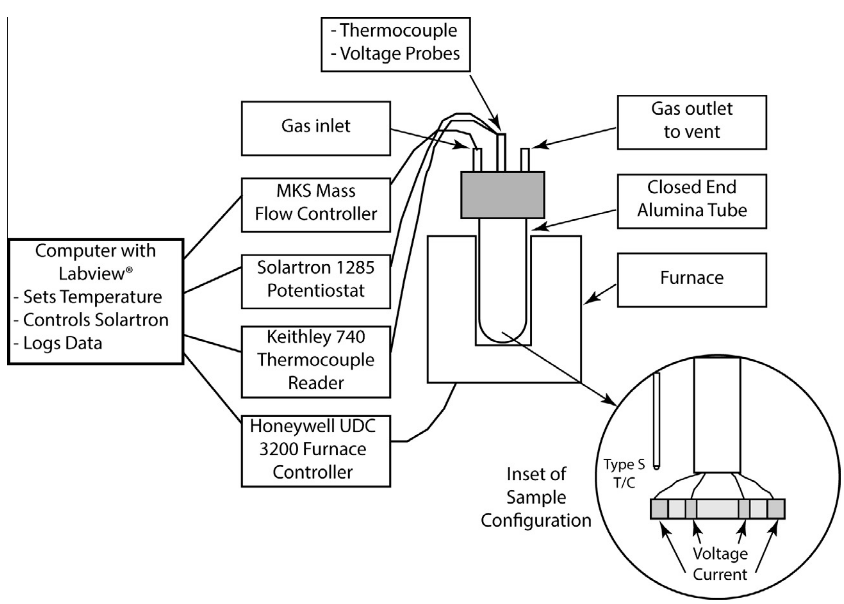

Fig. 1. Versatile automated electrical resistance measurement system schematic. The system is used for 4-probe I–V measurements on either a bar sample, shown in the inset view, mounted in a closed-end alumina tube; or for 2-probe I–V measurements on a disc sample mounted in an open-ended alumina tube. By adding an automated relay to the system, simultaneous 2/4-probe I–V measurements can be made on a disc sample with centrally located 4-probe potential contacts.