Fabrication of In-situ SiC/C Thermocouples by Selective Area Laser Deposition

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Abstract

With the intrinsic nature to process relatively small features, selective area laser deposition (SALD) is a potential technique to fabricate complex shaped macro-components with in-situ high-resolution micro-devices. In this study, SALD was used to deposit in-situ silicon carbide/carbon (SiC/C) thermocouples on alumina and silicon carbide substrates with a CO_2 laser. Tetramethylsilane (TMS) and acetylene (C_2H_2) were chosen as precursors for deposition of the silicon carbide and carbon lines respectively. The electromotive force (emf) of the deposited thermocouple was measured and found to respond sensitively to temperature variations from room temperature to 800^0C. The effect of the deposition parameters on the product morphology was also investigated.

1 Introduction

Among the many advantages of Solid Freeform Fabrication (SFF) technology is the possibility of building geometrically complex shapes, tailoring functionally graded materials or composites spatially, and embedding electromechanical devices in situ within parts during the fabrication process. An approach to these goals is to combine two SFF methods, Selective Area Laser Deposition (SALD) and the closely related SALD-Vapor Infiltration (SALDVI)[1-5]. SALDVI is used to build the bulk structure of the part while SALD is used to embed the sensors. Many kinds of sensors, such as thermistors, thermocouples, strain gauges, etc., could be formed based on this approach. Since SALD and SALDVI are completely compatible gas phase approaches, whole parts can be fabricated in a single integrated SALD/SALDVI process system. As a first attempt using the SALD/SALDVI technique, thermocouples have been embedded within a silicon carbide substrate. This paper discusses the SALD formation of the thermocouple; the SALDVI substrate is described elsewhere in these proceedings [6].

Forming a good thermocouple requires consideration of several factors including compatibility with the substrate, formation of a strongly bonded hot end junction, connections from the cold end to readouts, and the physical stability. To avoid thermal residual stresses, differences in the thermal expansion coefficient between the substrate, insulation, and thermocouple lines must be as small as possible. Obviously, embedding SiC/C thermocouples into a SiC substrate has the advantage of the low thermal expansion mismatch, which will help to maintain continuity of the thermocouple lines during heat/cool cycles.

The first SiC/C thermocouple was developed to measure the temperatures of molten steel by Fitterer in 1933[7]. This thermocouple was formed through simply putting SiC and graphite rods together. An extremely high electromotive force constant, up to $300\mu V/^0C$, was reported. But, this thermocouple is not stable in both liquid metals and gas environment. This greatly hindered the further application of SiC/C thermocouple. With the SALD technique, these problems could be avoided by enclosing SiC/C thermocouples within a SiC substrate.

2 Experimental procedures

2.1 Procedures of the device fabrication

An overall process schematic to make the embedded thermocouple device is shown in Figure 1. The substrate is first built layer by layer with SALDVI. An electrically insulating layer is then put on this substrate. With different gas precursors, the thermocouple lines are deposited on the insulation layer by SALD. The final steps of building device are repeating the electrically insulating layers and continuing to build the remainder of the substrate with SALDVI. Single-point or multiple-point thermocouples could be made in one layer through this process. In addition, multiple thermocouples could also be built within different layers. This will be useful when the multi-point temperature control is needed.

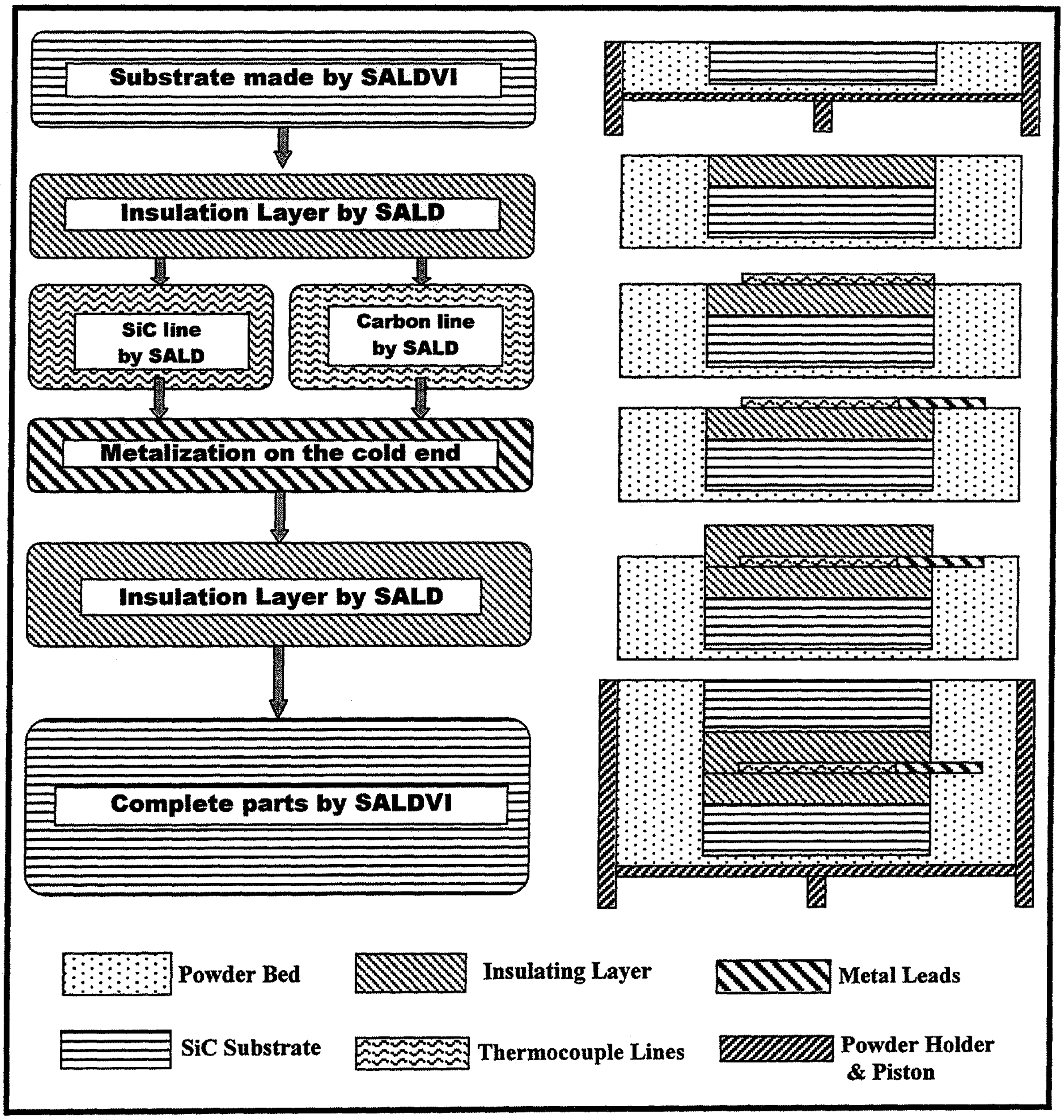

Figure 1 Flowchart of fabricating in-situ device embedded in macro-component

2.2 Deposition of silicon carbide line, carbon line and insulation layer

SALD was carried out using a SFF system which is comprised of a reaction chamber, powder delivery system, 2-D laser beam scanning control stage, gas precursor supply system, observation and recording system, closed-loop temperature control system, and a CO_2 laser with a maximum output of 50W in cw mode and a wavelength of 10.64μm. To get a theoretical spot size of about 500μm, a 254mm focal lens was used. Maximum power density is about 255W/mm^2.

Tetramethylsilane $Si(CH_3)_4$ (TMS) was chosen as the precursor for deposition of SiC because it produces silicon carbide free from halogens and the by-products of pyrolysis reaction are neither corrosive nor contaminant.

For carbon deposition, several precursors, acetylene, ethylene, and methane, were evaluated. The overall reactions are as followings:

$$C_2H_{2(g)} \Leftrightarrow 2C_{(s)} + H_{2(g)}$$
$$C_2H_{4(g)} \Leftrightarrow 2C_{(s)} + 2H_{2(g)}$$
$$CH_{4(g)} \Leftrightarrow C_{(s)} + 2H_{2(g)}$$

Insulation layers were deposited with a gas mixture of TMS and NH_3. Preliminary thermocouple depositions were carried out on alumina and silicon carbide substrates, which were made with conventional sintering method. Additional experiments were conducted on SALDVI silicon carbide substrates.

2.3 Measurement of electromotive force (emf)

The emf of the thermocouple was measured by connecting the cold end directly to the measurement unit through copper wires. Strawberry tree software was applied to collect the emf signals. The laser beam was used to heat the hot end of the thermocouple. A standard K type thermocouple was also put on the hot end as an independent temperature sensor to monitor the temperature change of the hot end. The measurement setup is shown in Figure 2.

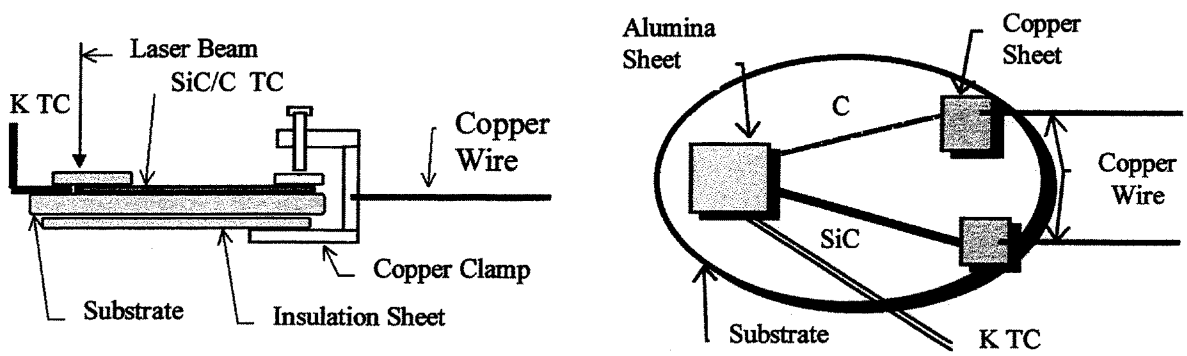

Figure 2 Setup for measuring emf of the SiC/C thermocouple

2.4 Characteristics of the deposited product

SALD thermocouple line morphologies were analyzed with scanning electron microscopy (SEM). The thickness profile of the lines perpendicular to the scan direction was examined with a contact profilometer.

3 Results and discussion

3.1 Silicon carbide lines

A series of SiC lines were deposited on the sintered alumina, the sintered SiC substrates and SALDVI SiC substrates. It was found that line profiles were affected by the deposition conditions of temperature and gas pressure. Generally, with the increase of temperature and TMS gas pressure, the line shape changes from a sharp peak to a flat top to a coarse, rough top surface. In addition, both width and thickness increase with temperature and gas pressure at the range of the present research. These results are listed in Table 1; the typical profiles of deposited lines are shown in Figure 3.

Table 1 Width and thickness of deposited silicon carbide lines on the alumina substrate (scanning speed: 0.5mm/sec; 8 passes)

TMS gas pressure	Line width And Thickness (μm)	SALD Temperature (^{0}C)				
		700	900	1000	1100	1250
20 Torr	Width	/	510	620	750	1040
	Thickness	/	13.9	28.8	42.1	58.3
40 Torr	Width	/	390	570	810	1195
	Thickness	/	31.9	114.1	142.9	143.8
60 Torr	Width	375	410	/	830	/
	Thickness	16.5	68.8	/	147.8	/

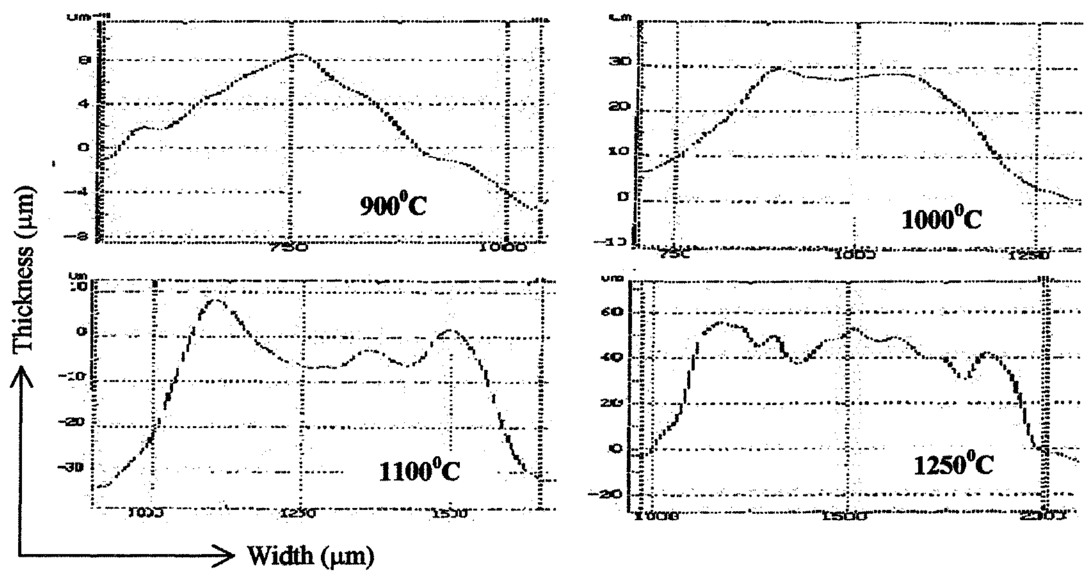

Figure 3 Typical profile changes of the deposited SiC lines (P_{TMS}=20 Torr; Scanning speed: 0.5mm/sec; CO_2 laser; Alumina substrate; 8 passes)

3.2 Carbon deposition

The deposition rate of carbon is relatively slow compared to that of silicon carbide especially when methane or ethylene was used. This is consistent with other research results [8]. After the preliminary experiments, acetylene was chosen for deposition of carbon lines. Three kinds of morphologies, cotton-like shape(Fig 4a), disc shape(Fig 4d), and nodular shape(Fig 4b&c), are observed. The cotton-like shape was observed usually at a low deposition temperature, while nodular morphology was obtained at relatively high temperatures. The disc shape was only observed on the SiC substrate. When the deposition temperature exceeds 1250°C on the both alumina substrate and SiC substrate, carbon growth becomes unstable. The similar phenomenon was also observed on the effect of the gas pressure. It was found that the deposition process could be easily controlled if the C_2H_2 pressure is lower than 200Torr. The surface quality of the deposited carbon lines depends on the surface finish of the substrate. In addition, depositing carbon on the alumina substrate is easier than depositing on the SiC substrate under the same deposition conditions when the CO_2 laser was used. These results showed that the processing conditions obviously affect the deposition of the carbon.

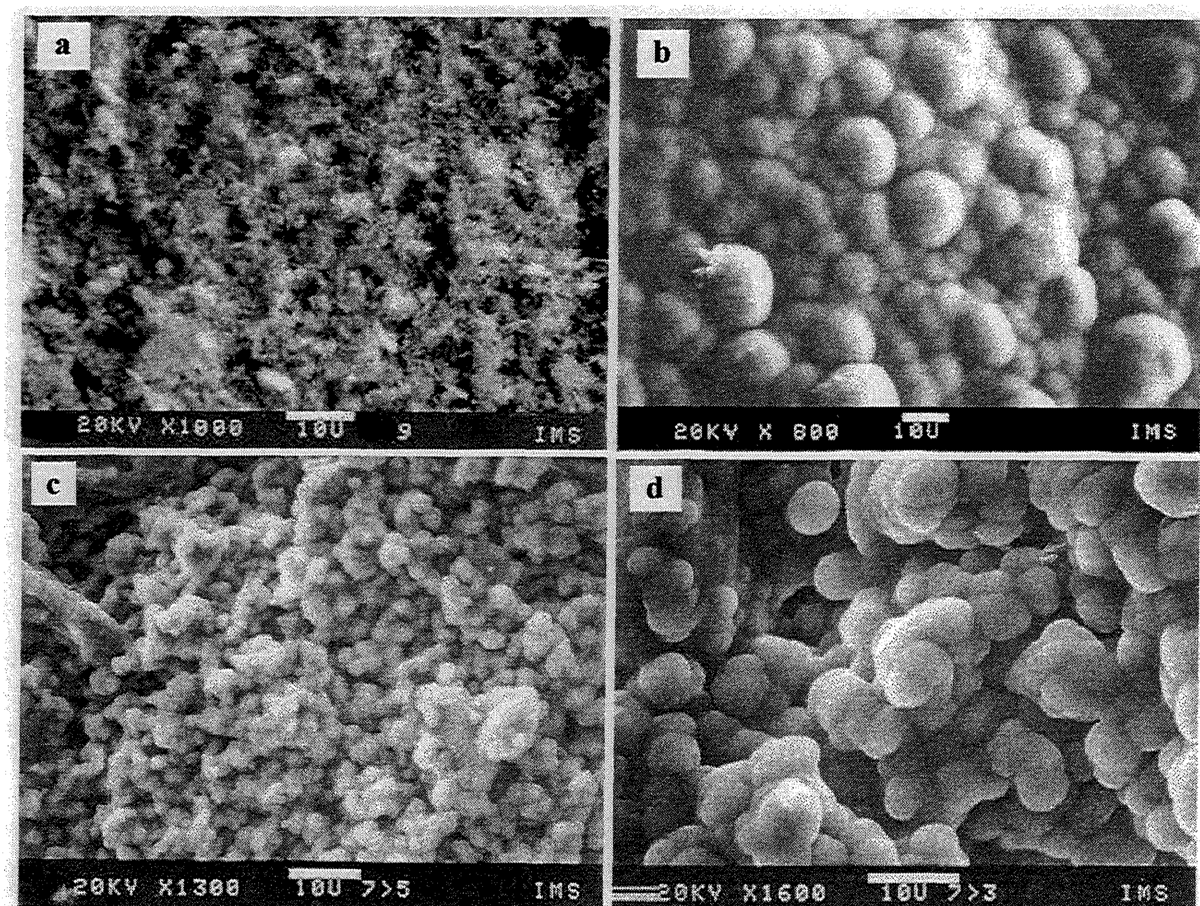

Figure 4 Surface Morphologies of the deposited carbon lines using CO_2 laser on
(a) P_{C2H2} = 200 Torr; Temperature: 800°C; Scanning Speed: 0.06mm/sec; Alumina substrate; 20 passes.
(b) P_{C2H2} = 100 Torr; Temperature: 1250°C; Scanning Speed: 0.76mm/sec; Alumina substrate; 20 passes.
(c) P_{C2H2} = 100 Torr; Temperature: 1000°C; Scanning Speed: 0.38mm/sec; SiC substrate; 20 passes.
(d) P_{C2H2} =200 Torr; Temperature: 900°C; Scanning Speed: 0.13mm/sec; SiC substrate; 20 passes.

3.3 Deposition of insulation layers

Since the thermocouple must be electrically insulated from the substrate, an insulating layer is deposited from the gas mixture of TMS and NH_3. It is found that the surface quality of the insulating layer depends on the scan speed and the surface finish of the substrate. For a substrate with poor surface finish, a very low scanning speed is required to get a uniform insulating layer. Under a condition of a 15Torr TMS plus 15Torr NH_3 and constant temperature (900^0C), a continuous layer with a thickness of about 30μm is obtained. Electrical measurement showed that it is an insulator.

3.4 Electromotive force from SiC/C thermocouples

Some physical parts with the SiC/C thermocouples are shown in Figure 5. The electrical

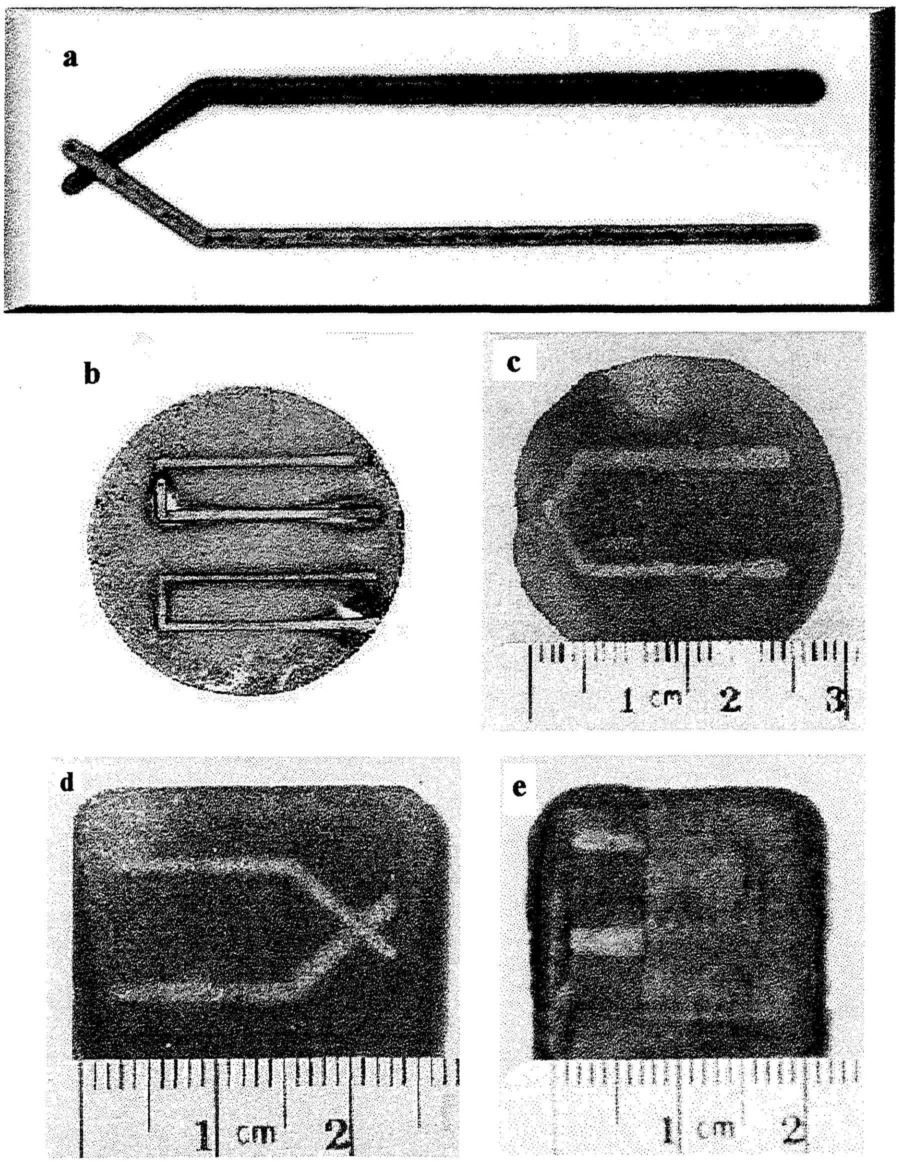

Figure 5 Physical parts with the thermocouples on (a) alumina substrate; (b) the sintered SiC substrate; (c) the single layer SALDVI SiC substrate; (d) multiple-layer SALDVI SiC substrate; (e) multiple-layer SALDVI SiC substrate with the insulating layers at the bottom and top of the thermocouple.

measurements showed that the SALD SiC/C thermocouples, which were deposited on both sintered alumina substrates and SALDVI SiC substrates, exhibited sensitive response to temperature variation. Two typical temperature-emf curves from the SiC/C thermocouples on alumina and SiC substrates are given in Figure 6.

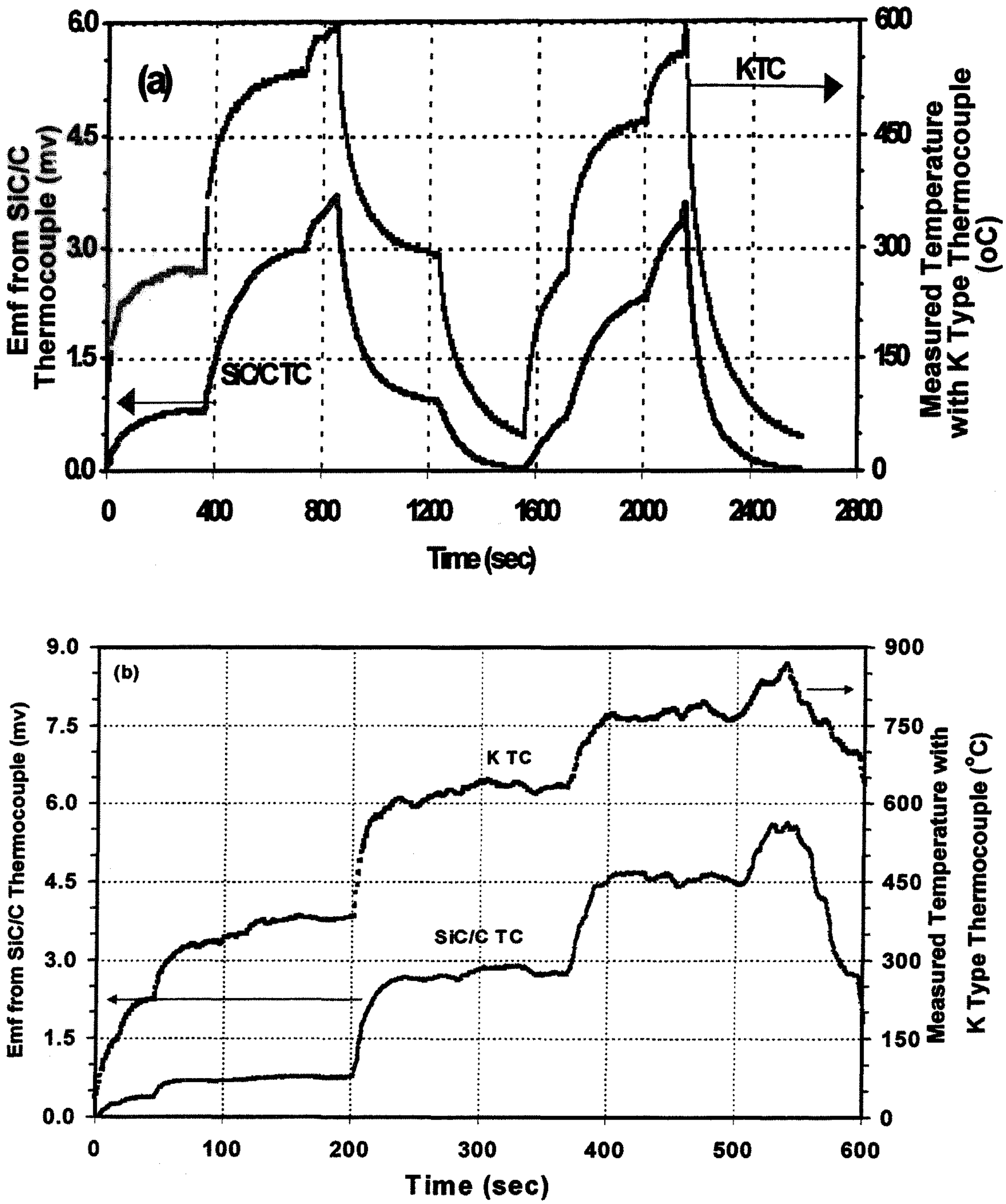

Figure 6 Emf from SiC/C thermocouples as a function of temperature: (a) Thermocouple deposited on sintered alumina substrate; (b) Thermocouple deposited on SALDVI substrates.

It was found that the electromotive force constants are almost same for both thermocouples on the alumina and SiC substrates, which is around 6.7μV/°C. This value is much lower than Fitterer's results [7]. This difference probably comes from a difference in material structures. First, the SALD carbon line may not be in fully graphite form. In addition, the electrical conductivity of silicon carbide may also be too low. The experiments have shown that SiC obtained is amorphous and non-conductive when it is deposited at low temperature and with an unfocused beam, while the SiC substrates made with the conventional sintering method showed a fully conductive behavior. These indicated that the low emf constant could be increased through optimization of SALD process to obtain better structural characteristics of the deposits.

4 Summary

The preliminary experiments have shown that it is feasible to make a structure containing in-situ thermocouples by a combined SALDVI and SALD approach.

Silicon carbide lines, carbon lines and insulating layers have been deposited from the precursors TMS, acetylene, and a gas mixture of TMS and ammonia respectively. The line profile of silicon carbide changes with temperature and gas pressure. Three type of carbon morphologies, cotton-like shape, disc shape and nodular shape, are identified.

It was found that the SALD SiC/C thermocouples respond to temperature variation sensitively. The emf constant of SALD thermocouple is much lower than that obtained with the SiC/C thermocouple made of SiC/C rods. Optimization of SALD process is needed to improve the emf constant of the in-situ SiC/C thermocouple.

Acknowledgement

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