Effect of Ramp Rate on Heat Generating Portion of SiC Heating Elements

Kavitha S D, Shivanna D M, Sunil Kumar Kulkarni, R Suresh

Abstract— In the present work silicon carbide is used in electrical resistance heating elements because of its high hardness, corrosion resistance, abrasion and high thermal conductivity. This material exhibits outstanding properties at high temperatures up to 1600°C, either in oxidizing or reducing atmosphere condition; the creep and strength is very high when compared to other materials. Silicon carbide heating element consists of a hot zone with a cold end. A range of sintered porous silicon carbide heating elements are prepared and analyzed. The results indicate that the push rate influenced the electrical resistance of the heat generation portion of heating element and saving of energy consumption with increase of production rate.

Index Terms— Hot Zone, Heating Elements, Resistance, Silicon Carbide, Sintering.

_ _ _ _ _ _ _ _ _ _ _ _

1 INTRODUCTION

SILICON Carbide is polymorphic in nature and crystallizes at room temperature and atmospheric pressure into a diamond lattice. The basic structure of silicon carbide is bonded together by strong hybrid bonds and highly directional, composed of tetragonal units of silicon and carbon atoms. The silicon carbide bonds are essentially covalent.

The impurities affect the stability of any polytype but it depends mainly upon temperature for its formation. β -silicon carbide is formed at 525°C and is stable up to 2100°C, beyond which it begins a slow transformatiom to α -silicon carbide at 2100°C, process is rapid at 2300°C and completes at this temperature, [1]. The electrical resistance of silicon carbide varies considerably when sintered in nitrogen atmosphere, [2]. Recrystallized silicon carbide is a porous constitute 20% of the structure. The theoretical density of recrystallized silicon carbide is approximately 2.9-3 g/cc and its strength is about 90 MPa, [3].

In powder compacts the surface of the grains is convex, while sharp concave surfaces are formed at the contact points between the grains. The difference in the vapour pressures between concave (low pressure) and convex (high pressure) surfaces of the silicon carbide particles is in the order of 10-6 to 10-5 atmospheres [4][5].

Silicon carbide has been recognized as a high performance material for structural ceramics because of its unique combination of properties, such as high mechanical properties maintained at high temperature, high wear resistance, high thermal conductivity and low electrical resistivity [6],[7],[8],[9],[10],[11]. Silicon carbide is widely used as electrical heating element, in spite of the electrical ageing (i.e. increase of electrical resistivity) and non-linear electrical resistivity variation with running time and temperature [12],[13][14],[15]. They have different sections in which the central heat generation zone is called the hot zone and other two end segments are called cold zone [16],[17],[18],[19].

2 METHODOLOGY

2.1 Preparing Ingredients and Blending:

Variable Grit size of green silicon carbide.

2.2 Extrusion:

Lumps after mixing ingredients in a sigma mixing machine is placed in pressing machine then pressed into any required shape.

2.3 Drying:

After forming, it is dried at $20 \sim 40$ °C for 24 hours at room temperature, then dried at $150 \sim 180$ °C for 3 -4 hours depending on the diameter of element.

2.4 Sintering:

Dried extruded silicon carbide heating elements are sintered in high temperature continuous graphite tube furnace. Fig. 1 shows the flow chart for processing SiC heat generating portion of heating element.

2.5 Testing:

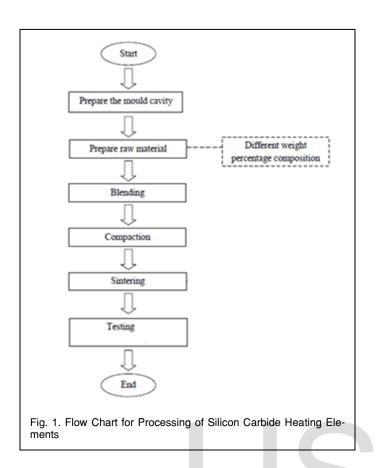
Sintered silicon carbide heating elements are tested for their electrical resistance by passing current with controlled voltage.

Author Kavitha S D is currently pursuing PhD program in Dept. of Mechanical Engineering, BTLIT M, VTU University, Bangalore, India. E-mail: kavitha88.dms@gmail.com

Co-Author Shivanna D M, DMS Heating Elements, Bangalore, Karnataka-Indi. E-mail: <u>dmshivu@gmail.com</u>

Co-Author Sunil Kumar Kulkarni, Professor, Dept. of Mechanical Engineering, ACE, Bangalore, Karnataka-Indi.
E-mail: suneelkumarkulkarni@rediffmail.com

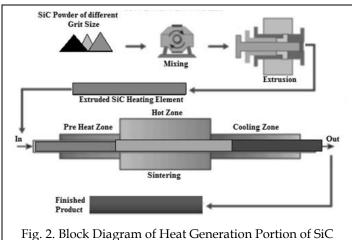
Co-Author R Suresh, Professor, Dept. of Mechanical Engineering, M.S.R university of Applied Sciences, Bangalore-India. E-mail: <u>sureshchiru09@gmail.com</u>



3 EXPERIMENTATION

3.1 Preparing Ingredients and Blending

Silicon carbide with green silicon carbide suitable purity of 99% or more. The granularity is from 5 to 10% of coarse grains of 24 # to #36, 45 to 50% of medium grains of 80 # to 90 #, and fine grains of 1200 # or more account for 30 to 40%. The cellulosic material methylcellulose, preferred conditions for methyl cellulose are methoxy content 27-30%, viscosity range 3500-5600 mPa. s (at 20 ° C, 2%). These ingredients are mixed in a sigma mixing machine for a period of 45 minutes to 1 hour to form lumps by adding 8-9% of water. Samples of heat generation portion are produced as shown in Fig. 2.



Heating Elements

3.2 Extrusion

Lumps after mixing ingredients in a sigma mixing machine are placed in an extrusion machine and then pressed into the required shape (diameter and length) of hot zones and dried in oven.

Weigh green silicon carbide 24 # 1500 grams, 80 # 4500 grams, 300 # 1000 grams, 420 #1750 grams, 1200#3000 grams, dry mixed for 15 minutes, 1000 grams of methyl cellulose of 2% after mixing with water by 8-9% and mixing for 45 minutes, the blended lumps pressed at the pressure 85Mpa. Diameter of 25mm is extruded for the length of 500 mm.

3.3 Drying

Extruded tube shape elements are dried at $20 \sim 40$ °C for 24 to 48 hours at room temperature, then dried at $150 \sim 180$ °C for 3 -4 hours depending on the diameter of element.

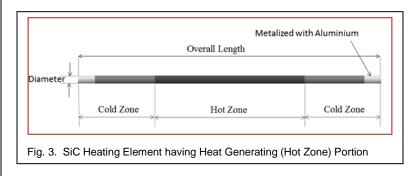
3.4 Sintering

Dried extruded silicon carbide heating elements are sintered in the continuous high-temperature carbon tube furnace firing. The furnace conditions are: preheating zone temperature 350°C-800°C, High temperature zone temperature of 2350°C -2450°C, carbon tube length 1950 mm; advancing variable pusher ramp speed by nitrogen protection.

3.5 Testing

Sintered silicon carbide heating elements are tested for its electrical resistance by passing current with controlled voltage. In this work Ø25mm silicon carbide heating elements have been produced by controlling the process parameters with controlled atmosphere.

After sintering, resistance has been measured and uniform glow of hot zone portion of silicon carbide heating elements. The standard resistance with attained allowable resistance tolerance of $\pm 15\%$ as tabulated below in table 1 to 4. The voltage supplied to the elements being tested, the current indicates based on elements porosity, uniform red hot evenness. Fig. 3 is the sample of SiC Heating Element having Heat Generating (Hot Zone) Portion.



| 1 | 1 | 0 |
|---|---|---|
| | | |

| | Г | TABLE 1 | | | |
|---------------------------------------|---------|---------|--------------|--|--|
| RESISTANCE OF HOT ZONE DIAMETER 25MM, | | | | | |
| LENGTH 500MM, PUSHER RATE 24MM/MIN. | | | | | |
| Sample | Voltage | Current | Resistance (| | |

| ···· 1 | 0 | | Resistance (Ω) | |
|--------|-----|-------|-------------------------|--|
| | (V) | (A) | Resistance (S2) | |
| 1 | 50 | 53 | 0.943 | |
| 2 | 50 | 51 | 0.980 | |
| 3 | 50 | 49 | 1.02 | |
| 4 | 50 | 52 | 0.961 | |
| 5 | 50 | 49.11 | 1.01 | |
| 6 | 50 | 52.18 | 0.958 | |
| 7 | 50 | 48.1 | 1.039 | |
| 8 | 50 | 48.55 | 1.029 | |

TABLE 2RESISTANCE OF HOT ZONE DIAMETER 25MM,LENGTH 500MM, PUSHER RATE 22MM/MIN.

| Sample | Voltage (V) | Current (A) | Resistance (Ω) |
|--------|----------------|----------------|-------------------|
| 1 | 50 | 75.12 | 0.665 |
| 2 | 50 | 77.3 | 0.646 |
| 3 | 50 | 78.43 | 0.638 |
| 4 | 50 | 75.53 | 0.661 |
| 5 | 50 | 76.8 | 0.651 |
| 6 | 50 | 75.93 | 0.658 |
| 7 | 50 | 74.95 | 0.667 |
| 8 | 50 | 77.15 | 0.648 |

From the Tables 1 to 4, the measured resistivity for an overall length of hot zone of different samples tested at temperature range from 30°C to 900°C as tabulated in Table 5 by varying ramp rate. And Table 5 is a value of resistivity Ohm per cm, temperature ° C of sintered silicon carbide heating elements by passing through a high temperature continuous graphite tube furnace at different speeds explained in results and discussion. The resistivity of the hot zone material sintered at various push rates ranging from 18 mm/min to 24 mm/min at a furnace temperature of 2390°C to 2410°C.

| TABLE 3 |
|---------------------------------------|
| RESISTANCE OF HOT ZONE DIAMETER 25MM, |
| LENGTH 500MM. PUSHER RATE 20MM/MIN. |

| Sample | Voltage (V) | Current (A) | Resistance (Ω) |
|--------|----------------|----------------|-------------------------|
| 1 | 50 | 80 | 0.625 |
| 2 | 50 | 79.36 | 0.63 |
| 3 | 50 | 81.32 | 0.614 |
| 4 | 50 | 78.65 | 0.635 |
| 5 | 50 | 79.53 | 0.628 |
| 6 | 50 | 80.22 | 0.622 |
| 7 | 50 | 79.94 | 0.625 |
| 8 | 50 | 80.54 | 0.62 |

TABLE 4RESISTANCE OF HOT ZONE DIAMETER 25MM,LENGTH 500MM, PUSHER RATE 18MM/MIN.

| Sample | Voltage (V) | Current (A) | Resistance (Ω) |
|--------|----------------|----------------|-------------------------|
| 1 | 50 | 83.23 | 0.600 |
| 2 | 50 | 83.96 | 0.595 |
| 3 | 50 | 84.3 | 0.593 |
| 4 | 50 | 83.51 | 0.598 |
| 5 | 50 | 82.86 | 0.603 |
| 6 | 50 | 83.0 | 0.602 |
| 7 | 50 | 83.71 | 0.597 |
| 8 | 50 | 83.31 | 0.600 |

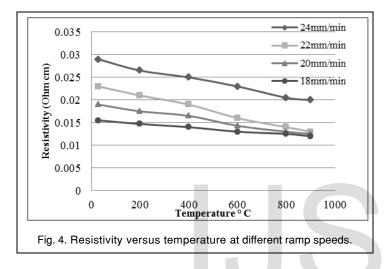
| TABLE 5 |
|--|
| RESISTANCE OHM (Ω).CM OF HOT ZONE DIA 25MM, LENGTH 500MM, |
| VARIABLE PUSHER RATE IN MM/MIN AT |

| Temp | Pusher Ramp Speed | | | | |
|-------------------|-------------------|-----------------------------------|----------|----------|--|
| erature | 24mm/min | 22mm/min | 20mm/min | 18mm/min | |
| in ^o C | | Resistance in Ohm (Ω).cm | | | |
| 30 | 0.029 | 0.023 | 0.019 | 0.0155 | |
| 200 | 0.0265 | 0.021 | 0.0175 | 0.0147 | |
| 400 | 0.025 | 0.019 | 0.0165 | 0.014 | |
| 600 | 0.023 | 0.016 | 0.0143 | 0.013 | |
| 800 | 0.0205 | 0.014 | 0.013 | 0.0125 | |
| 900 | 0.02 | 0.013 | 0.0125 | 0.012 | |

4 RESULTS AND DISCUSSION

In order to investigate the effects of hot zone recrystallization time, a continuous graphite tube furnace was used. By varying the push rate through the furnace, the duration at the sintering time can be varied thereby controlling the push rate. The faster the push rate, the shorter the sintering time and similarly the lesser the push rate longer is the sintering time.

Fig. 4 is a plot of resistivity versus temperature for hot zone of heating element produced from green silicon carbide of constant grain size formed by passing through a continuous graphite tube furnace at different pusher speeds.



The resistivity of the hot zone portion of silicon carbide material varies at various push rates from 24 mm/min to 18 mm/min at a constant sintering furnace temperature range of 2390-2410°C. A push rate of 18mm/min possesses minimum resistivity. Fig. 1 shows a graph of resistivity of hot zone of heating portion material versus temperature when sintered at different push rates. The reduction in resistivity achieved by slowing the push rate from 20mm/min to 18mm/min is small compared with that when the push rate is reduced from 22mm/min to 20mm/min. Although the push rate of 18mm/min showed the highest reduction in resistivity, such a slow push rate limits the production. The rate of production can thus be made the duration of sintering temperature and bathing time. With the particular furnace used, a push rate of 20mm/min was considered optimum.

5 CONCLUSION

The following conclusions were drawn;

The present work is aimed to provide a hot zone portion of silicon carbide electric heating element and a production process, which has the advantages of high resistance value stability, good quality performance, shortening the production process cycle and improving the yield of the silicon carbide electric heating element. rate of 18mm/min - the highest reduction in resistivitylimits the production. The rate of production can thus be made the duration of sintering temperature and bathing time. With the particular high temperature continuous graphite tube furnace used, a push rate of 20 mm/min was considered optimum. This saved the energy consumption of furnace and improved the production rate by reducing push rate to 120mm/hr. This also saved energy due to lower resistance of hot zone of heating element to generate high temperature upto 1600°C in the furnace.

REFERENCES

- [1] Baumann, "The relationship of alpha and beta silicon carbide", J. Elect. Chem. Soc., 99, 109-144.1952.
- [2] Forrest C.W, "The fabrication and properties of self bonded silicon carbide bodies", Special Ceramics 5. Proceedings of the fifth symposium on Special Ceramics held by The British Ceramic Research Association. Ed: Popper, p. 99-124, 1972.
- [3] Sunil Dutta, "Sinterability, strength and oxidation of alpha silicon carbide powders". J. Mat. Sci., 19, 1307-1313, 1984.
- [4] Kriegesmann. J, "Sintering phenomena in silicon carbide". Powder Metallurgy International, 18, (5), 341-343, 1986.
- [5] Sunil Dutta, "Improved processing of alpha-SiC". Adv. Ceram. Mat., 3; 257-62. Lewis Research Center, Cleveland, OH 44135, 1988.
- [6] Styrbjorn Amberg, "Sintered electric resistance heating elements and methods of producing such elements," US patent US3051924 (A), August 28,1962.
- [7] Gustav Schrewelius Nils, "Electric resistance elements of silicon carbide and metal silicide," US patent US3246275 (A), April 12, 1966.
- [8] Carlson Ronald J, "Electric resistance heater and method of making," US patent US3404202 (A), Dec 01, 1968.
- [9] Hierholzer Jr Frank J, "Method for producing improved silicon carbide resistance elements," US patent US4426405 (A), Jan 17, 1984.
- [10] Watenabe, "Process for Producing Silicon Carbide Heating Elements," US Patent US 4336216 A, June 22, 1982.
- [11] Frank J, "Silicon carbide resistance element," US Patent 4443361 A1, April 17, 1984.
- [12] Kondo Akira, "Production of silicon carbide heating element," Japan Patent JPH0692733 (A), April 5, 1994.
- [13] Kitahama Hiroaki, "Silicon Carbide Heating Element", Japan Patent JPH09213462 (A), August 15, 1997.
- [14] Kitahama Hiroak, "Dense Sic Heater and Manufacturing Procedure for It," Japan Patent JP2001110553 (A), April 2001.
- [15] Liu Zhenlin, "Electrothermal Silicon carbide elements and its production Process," China Patent CN 1371235 A, Sep 25, 2002.
- [16] Gao Jiqiang, "Preparation Technique for Silicon Carbide Heating Elements," China Patent CN 1293020 C, JAN 3, 2007.
- [17] Odaka Fumi, "Method for producing silicon carbide sintered body for heater," Japan Patent JP1717216 (A1), Nov 02, 2006.
- [18] Beatson, "Silicon carbide heating elements," European Patent EP1645168 B1, Feb.21, 2007.
- [19] Li Qingkui, "Production Method for Silicon Carbide Composite Electric Heating Elements", China Patent CN 103274690 A, Sep. 4, 2013.

The reduction in resistivity achieved by slowing the push