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CRADA Final Report  
for  
CRADA Number ORNL93-0244

SCALE-UP OF MICROWAVE NITRIDATION  
OF SINTERED REACTION BONDED  
SILICON NITRIDE PARTS

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**MASTER**

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Lockheed Martin Energy Research Corporation  
for the  
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## CONTENTS

ABSTRACT .....	1
OBJECTIVES .....	1
PURPOSE, BACKGROUND, AND BENEFITS .....	2
TECHNICAL RESULTS .....	2
SUMMARY.....	6
PUBLICATIONS .....	7
DISTRIBUTION.....	17

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## ABSTRACT

Scale-up studies were performed in which microwave heating was used to fabricate reaction-bonded silicon nitride and sintered reaction-bonded silicon nitride (SRBSN). Tests were performed in both a 2.45 GHz, 500 liter and a 2.45 GHz, 4000 liter multimode cavities. The silicon preforms processed in the studies were clevis pins for diesel engines. Up to 230 samples were processed in a single microwave furnace run. Data were collected which included weight gains for nitridation experiments, and final densities for nitridation and sintering experiments. For comparison, nitridation and sintering studies were performed using a conventional resistance-heated furnace.

## OBJECTIVES

The objectives of the CRADA were:

- 1 - Golden Technologies would fabricate engine parts with their standard procedures. Approximately 500-1000 samples would be fabricated under this task.
- 2 - ORNL would fabricate microwave furnace insulation packages to nitride 100-200 samples at a time.
- 3 - ORNL would nitride the engine parts using about four different conditions of time and temperature appropriate to obtain fully reacted articles.
- 4 - Golden Technologies would sinter the nitrided engine parts using their standard procedures.
- 5 - Characterization of the samples would be split between ORNL and Golden Technologies. ORNL would determine weight gain during nitridation for every part, perform x-ray diffraction of selected nitrided parts, measure fracture toughness by indentation/strength techniques of selected sintered specimens, determine the flexural strength of selected sintered specimens, examine selected samples by SEM, and perform x-ray diffraction on selected sintered samples. Golden Technologies would determine density of each of the sintered parts, will perform quantitative optical characterization of selected sintered specimens, determine the flexural strength at room temperature.

Most of the objectives of the CRADA were met. Engine parts were fabricated, nitrided and characterized. However, the parts were not sintered to high density. Unfortunately, Golden Technologies has stopped work in the area of sintered reaction bonded silicon nitride.

## PURPOSE, BACKGROUND AND BENEFITS

The major reason for the failure of the introduction of ceramics into many applications has been cost of components. This is most noticeable in the automobile engine industry where ceramics would provide lighter, more fuel-efficient and less polluting vehicles. Silicon nitride ceramics are the leading candidate materials for high temperature structural applications because of their combination of excellent strength, fracture toughness, wear resistance, thermal shock tolerance and high temperature properties. Development has traditionally been directed towards materials with superior strength, fracture toughness, wear behavior, and creep resistance with cost a secondary consideration. Consequently, traditional materials tend to be very expensive and are not competitive with metal parts on a replacement basis in many applications. Sintered reaction-bonded silicon nitride (SRBSN) is a cost-effective material that is receiving increased industrial interest for structural applications at temperatures  $<1200^{\circ}\text{C}$ . Compared to traditionally processed SRBSN ceramics, microwave processing can result in improved temperature uniformity, reduction of the total reaction time, and reaction in a more controlled environment around the parts.

Development of SRBSN materials for structural applications was being examined by ORNL and Golden Technologies. The ORNL work is being done under the Ceramic Technology for Advanced Heat Engines Project. The focus of that work involved up-scaling of the process to large batch sizes. Golden Technologies was up-scaling the SRBSN process using conventional heating practices.

This cooperative project was proposed as a joint development program between Golden Technologies and Martin Marietta Energy Systems (MMES), the Contractor. Cooperative work was of benefit to both parties. ORNL was able to assess up-scale the microwave nitridation process using a more intricate-shaped part that is designed for application in advanced heat engines. Golden Technologies gained access to microwave facilities and expertise for the nitridation of SRBSN materials.

### Technical Results - Set 1

Two sets of silicon preforms were sent by Gary Garvey of Golden Technologies to ORNL for microwave processing. The first samples received were die-pressed disc samples that were nominally 4.5 cm diameter and 0.9 cm thick. The second samples were extruded clevis pins (CP). Specifics about the clevis pin samples with conventional thermal processing have been discussed in the Ceramic Technology for Advanced Heat Engines Project Progress Report, W.B.S. 1.5.2.2, Feb. 1995, by Golden Technologies.

The binder burnout of the disc samples was done at a final temperature of  $550^{\circ}\text{C}$ . The binder burnout of the clevis pins was conducted at a final temperature of  $350^{\circ}\text{C}$ . Nitridation of the two sample types was conducted in both a graphite element furnace in flowing nitrogen, and in a 500 L, 2.45-GHz multimode microwave furnace. Test pieces were placed in graphite and BN crucibles for the nitridation treatments in the graphite furnace. Test samples were placed in BN and  $\text{Si}_3\text{N}_4\text{-SiC}$  crucibles for nitridation in the microwave furnace. Specifics about the composition of the  $\text{Si}_3\text{N}_4\text{-SiC}$  crucible have been discussed in previous reports. Two types of insulation configurations were used for the microwave nitridation treatments. The first insulation setup consisted of a central sample crucible, which was surrounded by a 2.5 cm thick layer of  $\text{Si}_3\text{N}_4\text{-BN}$  powder, all of which was enclosed in a 2.5 cm thick alumina fiberboard box. The second insulation setup consisted of 2.5 cm thick alumina fiberboard box, built directly around the sample crucible, with no intermediate powder layer.

The first three nitridation experiments were performed to provide base-line values of the percent weight gain for the disc and clevis pin samples as nitrided by conventional heating (conv.). Table 1 is a compilation of data and experimental conditions for these and other key nitridation experiments performed.

The first microwave scale-up experiment (MW), experiment 4, utilized the traditional microwave packaging system consisting of a BN crucible surrounded by powder and fiberboard insulation. The standard deviation of the percent weight gains, as seen in Table 1, was very good and this was supported by the uniform appearance of the samples. The average weight gain was nearly identical to the experiment 2, control. Fig. 1a shows the sample temperature-microwave power profile for this experiment. Note, the smooth temperature and power traces, and the increase in microwave power required after the nitridation reaction was completed. Experiment 5 was a scale-up to 21 disc samples using the same setup as experiment 4. In contrast to experiment 4, Fig. 1b shows the uneven temperature profile and the erratic microwave power control of this run. The average weight gain was slightly lower than the control, however the most important result was that two of the discs exhibited a slight amount of unreacted silicon metal on their surface and had approximately two percent less weight gain than the remainder of the discs. It appeared that the insulation system trapped too much of the heat released during the exothermic nitridation, resulting in sharp temperature excursions. As a result of this experiment, it was postulated that a more simple, less efficient insulation setup might be possible.

Several insulation setups were tested in an effort to develop an insulation arrangement excluding packing powder as the primary insulation. Clevis pin samples were used for these tests. The most promising tests were based on an experimental setup previously described in the Ceramic Technology for Advanced Heat Engines Project Progress Report, W.B.S. 1.1.2.4, June-July 1994. This experiment setup utilized a hybrid-heating,  $\text{Si}_3\text{N}_4$ -SiC crucible enclosed in an alumina fiberboard box. Experiment 6 was performed with this basic setup. Data from this test is shown in Table 1. 36 clevis pins were nitrided in this test. The average percent weight gain, 42.88, was comparable to the control value, 42.92. The standard deviation was 0.48. Sample appearances were very uniform. The total run time was 11 h, with a peak temperature of 1300°C. Figure 1c shows the uniform heating profile of this run. Note, the large reduction in microwave power which occurred during the exothermic nitridation of the silicon. Fig. 2 is a photo showing the sample package of experiment 6 with samples before and after processing. This experiment was repeated a second time with comparable results.

Future work planned for this CRADA included a scale up run with 100 or more clevis pin samples. A set up similar to experiment 6, using a  $\text{Si}_3\text{N}_4$ -SiC crucible appeared to be the best arrangement to use. Also, the results showed that slower heating rates will be required for success in nitridation.

### Technical Results - Set 2

Several activities were undertaken after the initial results were obtained: TGA analysis of clevis pin preforms were performed to determine their nitridation behavior in nitrogen; weight gain experiments involving silicon preforms were done in a graphite furnace to determine their nitridation behavior in  $\text{N}_2$  - 4 vol. %  $\text{H}_2$ ; microwave nitridation trials were performed; and x-ray diffraction analysis was performed on clevis pin RBSN processed by microwave and conventional heating.

The TGA study of clevis pin samples was undertaken in order to establish the temperature versus weight gain relationship for nitridation. The TGA study was performed using a TA Instruments SDT 2960 Simultaneous DTA-TGA. Data was collected for clevis pin and for comparison, bucket tappet and TM-145 silicon preforms. Flowing nitrogen was used for nitridation, since  $N_2 - 4 \text{ vol. } \% H_2$ , the preferred gas, could not be used with the Type "S" thermocouples (reaction problem). Fig. 3 is a graph showing the measured conversion of silicon to silicon nitride, as a function of temperature. The data in this graph is interpreted by drawing a vertical line between the data point on the percent conversion curve to the corresponding temperature (dotted line). The data indicate that the clevis pin material started nitriding at between 1000 to 1100°C and nitridation was complete at approximately 1400°C. The TM-145 sample and bucket tappet sample did not show significant nitridation until around 1250°C and were nitrided to approximately 95 % completion at 1450°C. The large difference in the nitridation behavior of the clevis pin and the other two sample types was due to the higher iron content of the clevis pin samples and possibly other compositional differences. The effect of various chemicals on silicon nitridation has been cited in the literature.

A series of nitridation runs was performed in a graphite furnace, in order to obtain the percent silicon conversion for the clevis pin and other comparison samples in  $N_2 - 4 \text{ vol. } \% H_2$ . The nitridation setup has been described in previous bimonthly reports. Fig. 4 shows that the percent conversion as a function of temperature for the clevis pin, TM-145, and bucket tappet samples. The nitridation of the clevis pins in  $N_2 - 4 \text{ vol. } \% H_2$  was approximately 40 % complete at 1200°C and was 95% complete by 1275°C. The nitridation of the TM-145 and bucket tappet samples was just starting at 1200°C, and was near completion at 1400°C. The absence of data points for the temperatures below 1200°C makes a comparison with TGA data at the start of the nitridation difficult, however this data does indicate that the addition of  $H_2$  appeared to lower the temperature at which conversion was complete for all samples. The effect of  $H_2$  additions on nitridation has been cited in the literature.

X-ray diffraction analysis was performed on selected samples to compare the  $\alpha$ -silicon nitride content of materials produced by conventional and microwave heating. Table 2 is a compilation of the data. High  $\alpha$ -silicon nitride values were obtained for clevis pin samples which were nitrided by microwave heating to a final temperature of 1300°C in nitrogen and in  $N_2 - 4 \text{ vol. } \% H_2$ . The slightly higher  $\alpha$ -silicon nitride content obtained for the sample processed in hydrogen was expected, based on previous work on the effect of  $H_2$  on nitridation. A comparable  $\alpha$ -silicon nitride content was obtained from a clevis pin preform processed to 1350°C in a graphite furnace in  $N_2 - 4 \text{ vol. } \% H_2$ . This appears to support belief that conventional and microwave processing follow the same nitridation routes. Clevis pin samples processed in a graphite furnace to 1450°C in  $N_2$  and in  $N_2 - 4 \text{ vol. } \% H_2$  showed much lower  $\alpha$ -silicon nitride contents, since the  $\alpha$ -silicon nitride is converted to  $\beta$ -silicon nitride as the sintering aids are converted to liquid phase at a temperature between 1350 and 1450°C.

In the initial work (Results-Set 1), several microwave insulation packaging setups were tested for the nitridation of clevis pins provided by Golden Technologies. The most promising setup for microwave processing of the clevis pins was a  $Si_3N_4$ -SiC crucible surrounded by a 2.5 cm thick rigid alumina fiberboard box. Two groups of 36 clevis pins were successfully nitrided using very fast heating cycles (<10 h) in this insulation arrangement. During the present semiannual period several experiments were done to test several factors concerning microwave nitridation of clevis pin preforms: effect of sample

number; the effect of sample spacing, touching versus spaced; the effect of alternative crucible construction; and the effect of lower H<sub>2</sub> content in the nitriding gas.

A summary of the results from various test runs made during this reporting period is presented in Table 3. Test 1 was a scale-up run to 100 test parts using the same insulation arrangement which had shown success in previous tests with 36 samples. In test 1 some sample melting occurred. Fig. 5 shows the temperature profile of test 1 including the thermal runaway which occurred. The same experiment was repeated, test 2, with the same results. The reason for the uneven processing in tests 1 and 2 is believed due to the much larger amount of heat released for 100 versus 36 samples. An alternative insulation setup, to the one used for test 1 and 2, was then built (Fig. 6) as a possible solution to the problem. This crucible arrangement consisted of Si<sub>3</sub>N<sub>4</sub>-SiC side panels for the side walls of the crucible and microwave-transparent hot-pressed BN setter plates for the top and bottom of the inner crucible. The BN top and bottom were added to absorb the excess heat released during nitridation. Tests 3 and 4 were performed to test this new crucible. Results of these tests in Table 3 show that both the exterior samples (samples 1-20, Fig. 6) and the interior samples (samples 21-36, Fig. 6) showed comparable nitridation. There was very minor distortion in a few of the exterior samples due to the fact that the crucible wall was cooler than the interior samples. Also, there was a problem with poor temperature control by the "Micristar" controller. Test 5 was performed to test the effect of reducing the amount of hydrogen on nitridation cycle. Good weight gain results were obtained in this test, indicating that lower amounts of hydrogen can be used, however the temperature oscillation still persisted. Test 6 was performed to test the effect of lowering the peak nitridation temperature to 1200°C, to avoid the controller problem. A lower average weight gain and a larger statistical deviation of weight gain values were obtained when the 1200 °C endpoint was used. It appears that a peak temperature above 1200 °C is necessary for complete nitridation of all samples. It is possible that a longer hold at 1200°C might dampen the sudden nitridation that occurs above 1200°C. Figure 7 is a plot of the temperature profile for test 6, which shows that the temperature of the exterior samples (1-20), as measured by a two-color pyrometer, initially lagged the temperature of the interior samples (21-36), as measured by a thermocouple, by up to 100 °C at 1100 °C and then moved closer as the temperature rose to 1200 °C. As conventional heating in nitrogen studies have shown, a peak temperature of 1300 °C is necessary for complete nitridation of all samples to occur. Test 7 was a scale-up to 100 clevis pins using microwave heating in a larger Si<sub>3</sub>N<sub>4</sub>-SiC-BN crucible, with the sample, edge to edge spacing at 0.5 cm, as was used in tests 3 and 4. Very good weight values and statistical deviation were obtained in this test, however as in previous tests, there was a problem with power control and also a slight warping of a few exterior samples. Test 8 was performed to see if increasing the packing density of samples (samples touching) would lead to better temperature control. Table 3 shows that a much better statistical deviation was obtained for this tight pack arrangement, but unfortunately there was partial sintering of some interior samples, as well as the problems with temperature control.

The results from the 8 tests have been used to determine the proper composition of sample crucibles, the proper spacing of samples, and the proper nitriding gas for microwave nitridation. Crucibles containing 60 wt % and 80 wt % SiC have been prepared for future nitridation tests. Gas containing at least 1 vol. % H<sub>2</sub> will be used, and sample spacing will be held at least 0.7 cm. The problem of inadequate microwave power control, which was experienced in some runs, will be addressed through the use of a state-of-the-art "Yokagowa" PID controller.

### Technical Results - Set 3

Several final experiments were performed to determine the optimum SiC content in the crucibles which were used for nitridation of silicon clevis pins (provided by Golden Technologies, Inc.). Three crucible types were made from gelcast plates containing 40, 60, and 80 wt % SiC, 4.2 wt % Y<sub>2</sub>O<sub>3</sub>, 0.8 wt % SiO<sub>2</sub>, and with a remaining balance Si<sub>3</sub>N<sub>4</sub> (crucible construction described in the Ceramic Technology for Advanced Heat Engines Project Progress Report, W. B. S. 1.1.2.4, April-May 1995). The final density of the crucibles was approximately 1.9 g/cm<sup>3</sup>, with the final nominal wt % compositions of the crucibles approximately the same as the gelcast plates. 81 clevis pins were weighed and then placed into each crucible at a spacing of 0.5 cm, edge to edge (Fig. 8). The samples were then heated in the ORNL 6 kW microwave furnace in nitrogen with 4 vol. % hydrogen to a final condition of 1300°C for 1 h. The total heating cycle of each run, excluding cool down, was 11.7 h. Samples were then weighed and weight gains were calculated.

Figure 9 shows the standard deviations of the weight gains of the total group of samples, the samples placed adjacent to the inside wall of the SiC crucible walls edge (outside samples), and the samples placed interior to the outside samples (inside samples). These data show that lowest standard deviations and the smallest deviations between the "outside" and "inside" samples were obtained with the "80 wt % crucible." The reason for this is that the microwave absorption and heat generated by of the "80 wt % crucible" resembled most closely the sample pieces, and this results in a more uniform temperature profile within the crucible. Figure 10 shows a photo of 90 clevis pins processed by microwave heating in a confirmation run using the "80 wt % crucible."

The final scale-up experiment performed for the Golden CRADA was a test to determine if the clevis pin samples could be processed in a horizontal layout and in multiple layers during both binder burnout and microwave nitridation on flat grooved setter trays. This processing method allows minimal handling of the samples and resembles the sample handling at Golden Technologies. Figure 11 is a drawing of the sample positioning on the "80 wt % SiC" setter plates. The samples and plates were heated to 400°C in air for binder removal, and then placed in an alumina fiber box in the microwave furnace where nitridation was performed according to the procedures used for previous crucible tests. The results of this last experiment were successful, with good weight gain measurements for all processed samples. The results indicate that this type of "easy" sample handling can be used in microwave scale-up processing.

### SUMMARY

Several statements are in order to summarize what has been learned to date from the Golden CRADA on the scale-up of the nitridation of silicon preforms and from the Ceramic Technology Project activities involving the scale-up of the nitridation and sintering of silicon preforms using microwave heating: Microwave heating shows promise for scale-up. Both silicon and RBSN do not suffer from "dielectric runaway," which plagues other ceramic-microwave systems. Microwave heating is attractive because it is one of the few ways to sinter SRBSN in a non-carbon environment. Microwave nitridation is a more attractive process than microwave sintering, because of the much more simple sample packaging used for nitridation (no powder insulation), and because the power required is less than half that required sintering. Microwave sintering of SRBSN is not viable for materials containing large amounts of contaminants, which may volatilize and migrate into the surrounding microwave insulation (rendering them useless) at temperatures above

1400°C. For both microwave nitridation and sintering processes, either special "hybrid-heating" crucibles must be used, or supplemental conventional heat must be provided to achieve uniform temperatures.

Commercialization of the microwave heating of SRBSN appears to be directed towards the nitridation part of the process and several companies have expressed interest. No further collaboration with Golden Technologies is planned at the present time.

## PUBLICATIONS

"SCALE-UP OF THE NITRIDATION AND SINTERING OF SILICON PREFORMS USING MICROWAVE HEATING" J. O. Kiggans, Jr., T. N. Tiegs, C. C. Davisson, M. S. Morrow and G. J. Garvey, to be published in Materials Research Society Proceedings (1996).

Table 1. Data for nitridation of Golden Technologies, Inc. silicon samples.

Exp #	Sample Type	Sample Total #	Furnace Type	Crucible Type	Run Time (hours)	Peak Temp. (°C)	Average % Wt. Gain	St. Dev. % Wt. Gain
2	disc	1	Conv.	BN	22	1450	44.70	N A
3	C P	1	Conv.	BN	19	1450	42.92	N A
4	disc	4	MW	BN	19	1450	44.67	0.04
5	disc	21	MW	BN	19	1450	44.21	0.38
6	C P	36	MW	Si <sub>3</sub> N <sub>4</sub> -SiC	11	1300	42.88	0.48

Table 2.  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> content of clevis pin samples nitrided by microwave and conventional heating.

Temp. (°C)	Furnace	Atmosphere	$\alpha$ -Si <sub>3</sub> N <sub>4</sub> Content (%)
1300	microwave	N <sub>2</sub> -4H <sub>2</sub>	90
1300	microwave	N <sub>2</sub>	87
1350	graphite	N <sub>2</sub> -4H <sub>2</sub>	87
1450	graphite	N <sub>2</sub> -4H <sub>2</sub>	58
1450	graphite	N <sub>2</sub>	9

Table 3. Summary of experimental test conditions and results for nitridation of clevis pins using microwave heating.

Test #	Sample Total	Crucible Type	Nitriding Gas	Run Time (h) / Temp. (°C)	Avg. % Wt. Gain Total Samples	Avg. % Wt. Gain Exterior Samples	Avg. % Wt. Gain Interior Samples	Standard Deviation of Total Avg. Wt. Gain
1	100	Si <sub>3</sub> N <sub>4</sub> -SiC	N <sub>2</sub> -4 vol % H <sub>2</sub>	8 / 1300	ND	ND	ND	ND
2	100	Si <sub>3</sub> N <sub>4</sub> -SiC	N <sub>2</sub> -4 vol % H <sub>2</sub>	8 / 1300	ND	ND	ND	ND
3	36	Si <sub>3</sub> N <sub>4</sub> -SiC-BN	N <sub>2</sub> -4 vol % H <sub>2</sub>	11 / 1300	43.33	43.26	43.43	0.23
4	36	Si <sub>3</sub> N <sub>4</sub> -SiC-BN	N <sub>2</sub> -4 vol % H <sub>2</sub>	11 / 1300	42.98	43.02	42.93	0.26
5	36	Si <sub>3</sub> N <sub>4</sub> -SiC-BN	N <sub>2</sub> -0.8 vol % H <sub>2</sub>	11 / 1300	43.49	43.50	43.49	0.14
6	36	Si <sub>3</sub> N <sub>4</sub> -SiC-BN	N <sub>2</sub> -0.8 vol % H <sub>2</sub>	10 / 1200	41.75	41.69	41.83	1.29
7	100	Si <sub>3</sub> N <sub>4</sub> -SiC-BN	N <sub>2</sub> -0.8 vol % H <sub>2</sub>	18 / 1300	44.18	44.18	43.74	0.32
8	60	Si <sub>3</sub> N <sub>4</sub> -SiC-BN	N <sub>2</sub> -0.8 vol % H <sub>2</sub>	16 / 1300	43.85	43.91	43.80	0.08

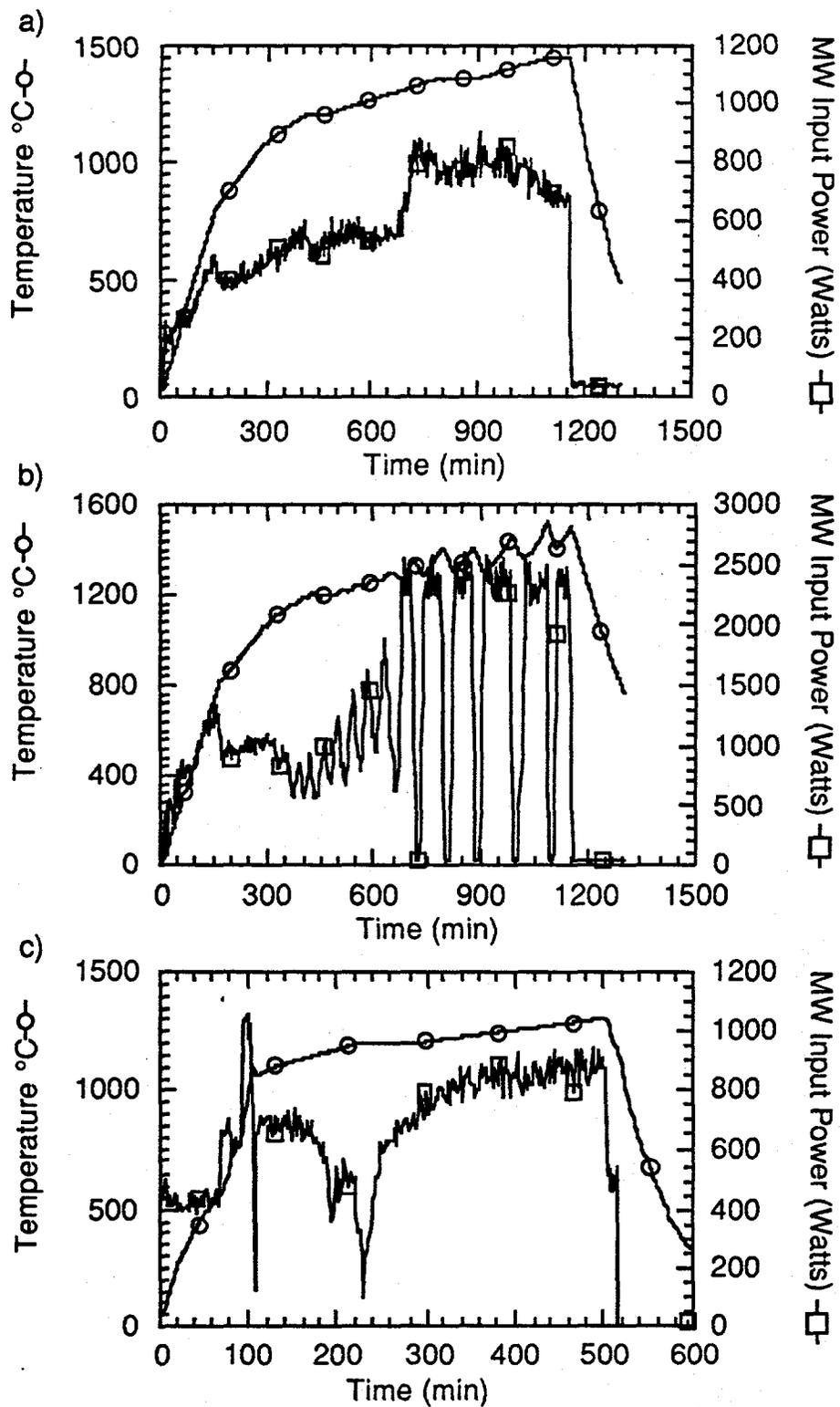


Fig. 1. Temperature-power profiles for experiments involving; a) 4 discs, b) 21 discs, and c) 36 clevis pins

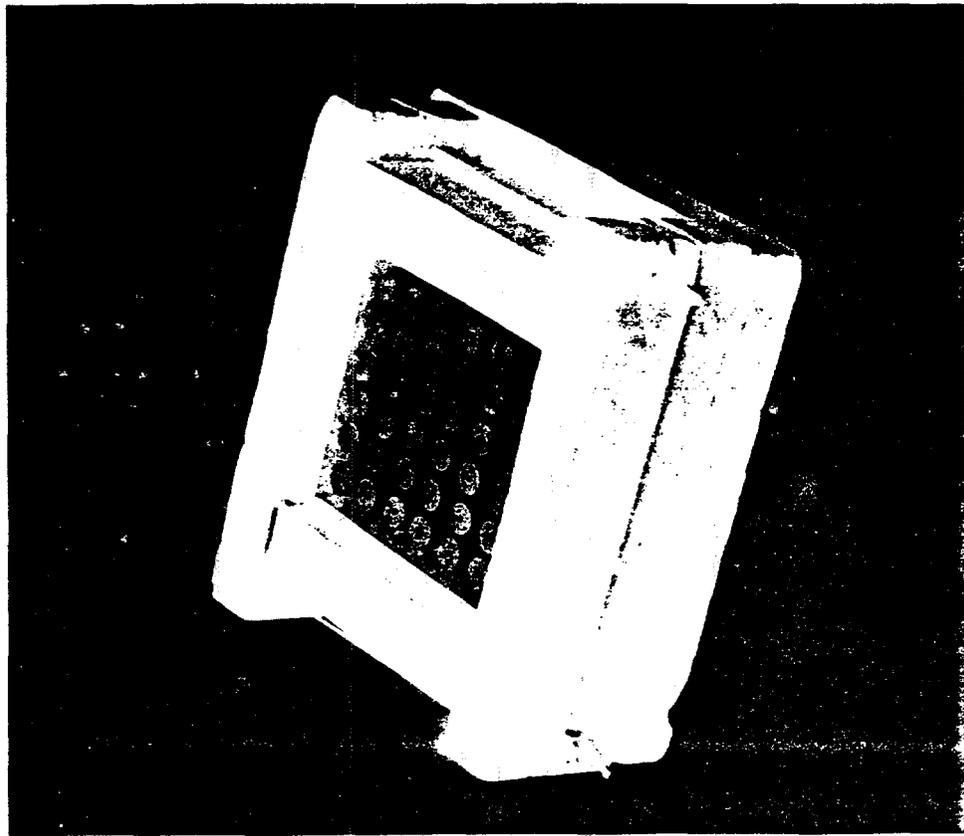
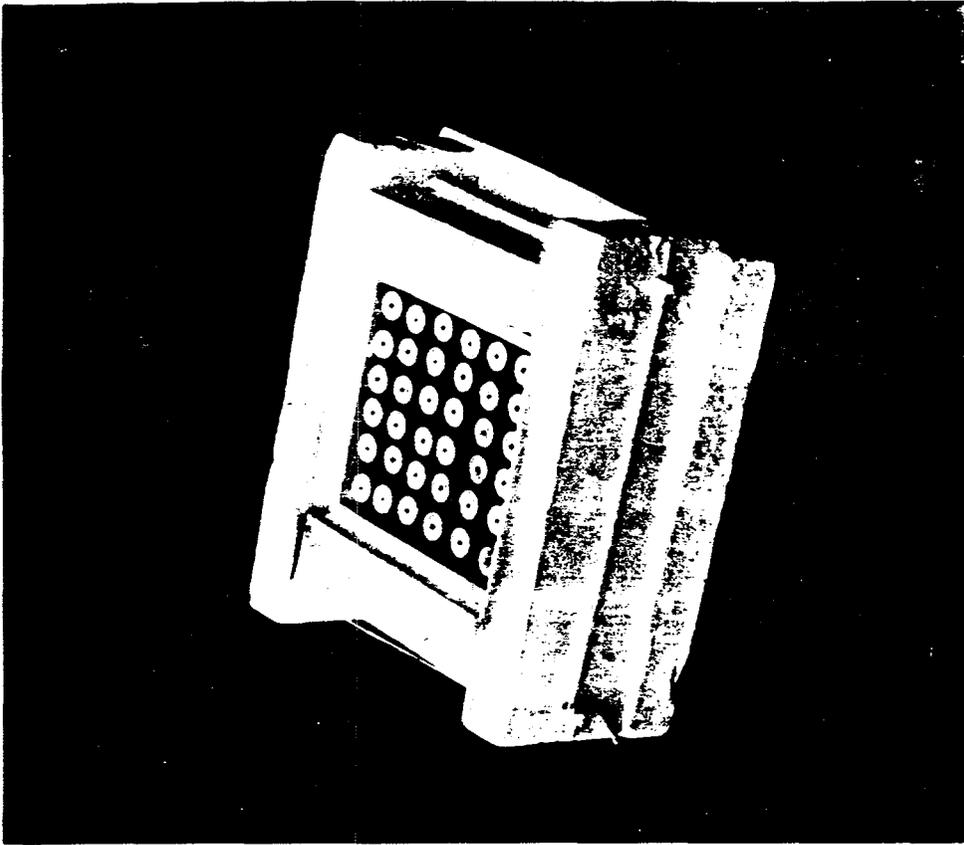


Fig. 2 Sample package for experiment 6 showing arrangement of clevis pins that had best nitridation results.

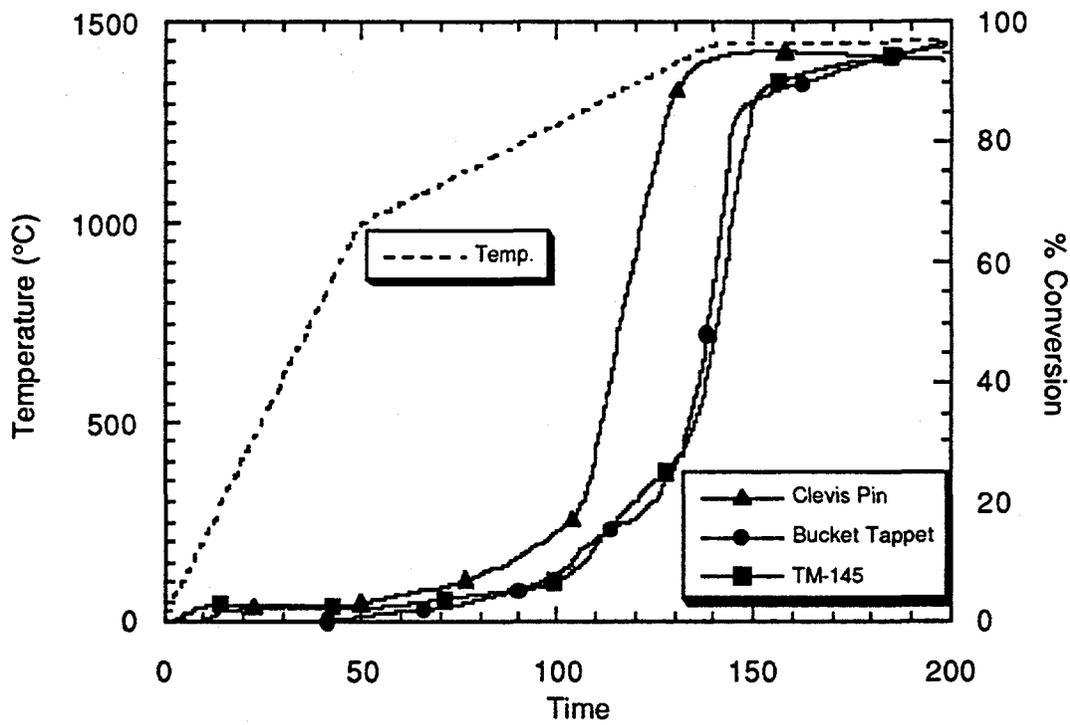


Figure 3. TGA analysis of silicon preforms.

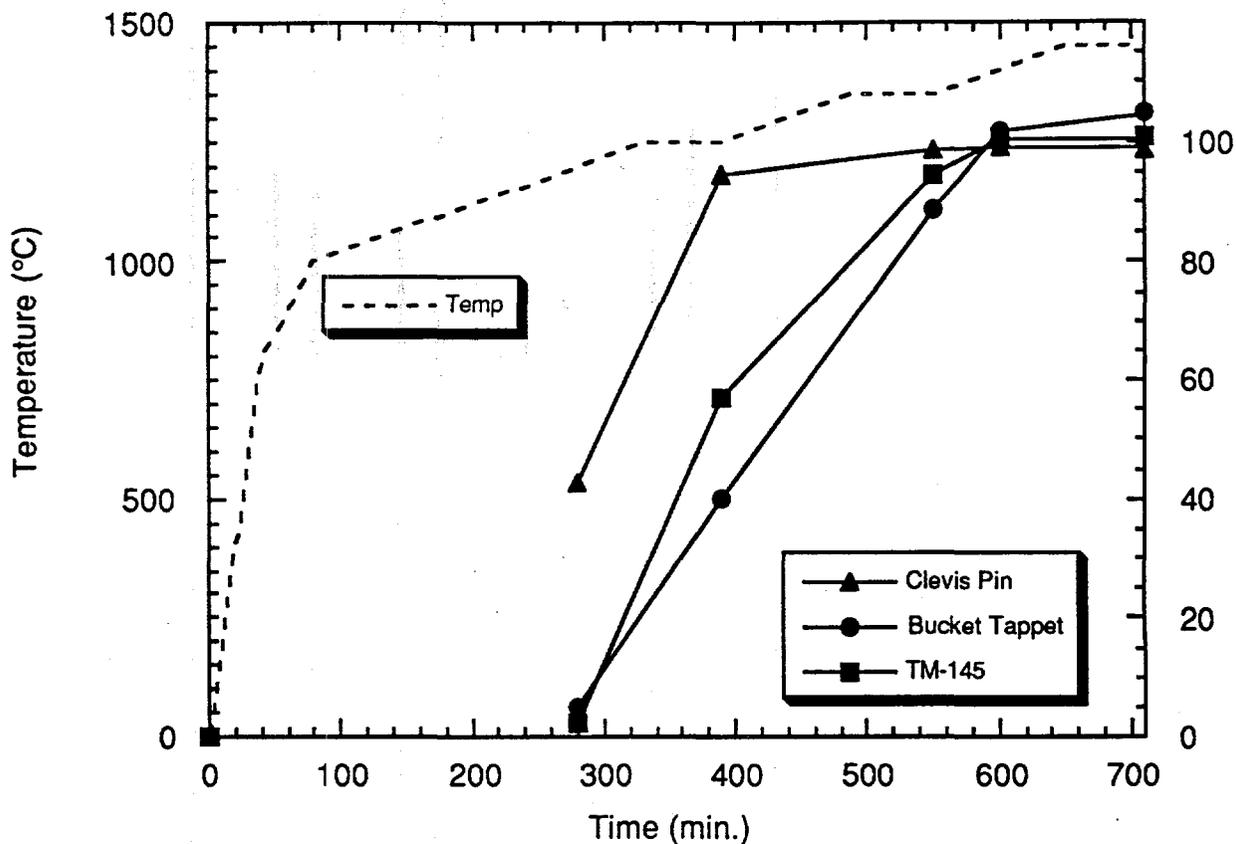


Figure 4. Percent Si-Si<sub>3</sub>N<sub>4</sub> conversion for samples nitrided in a graphite furnace in N<sub>2</sub> - 4 vol. % H<sub>2</sub>.

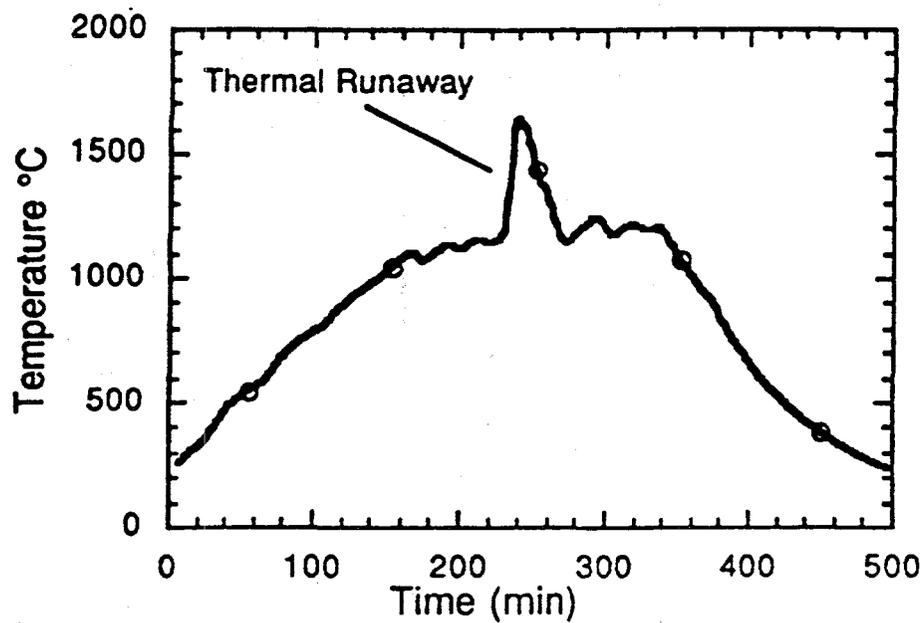


Fig. 5. Temperature profile for test 1 (Results Set 2).

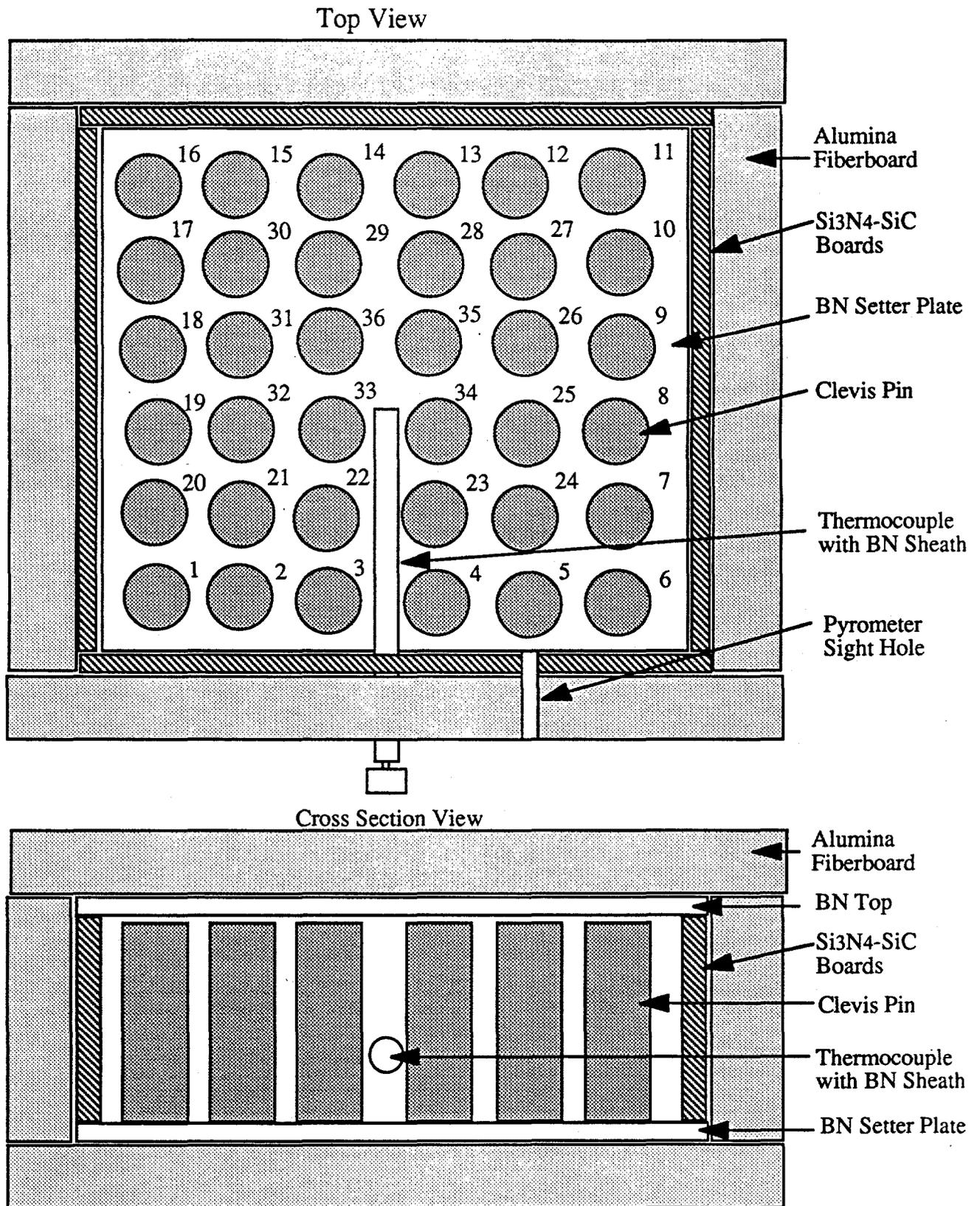


Figure 6. Alternate microwave package for processing silicon clevis pins

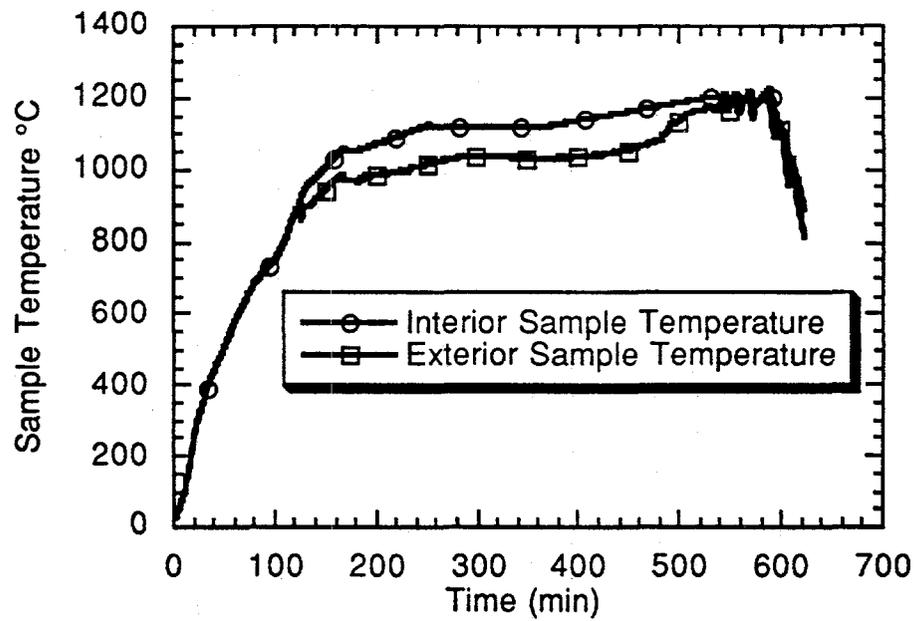


Fig. 7. Temperature profile for test 6 (Results set 2).

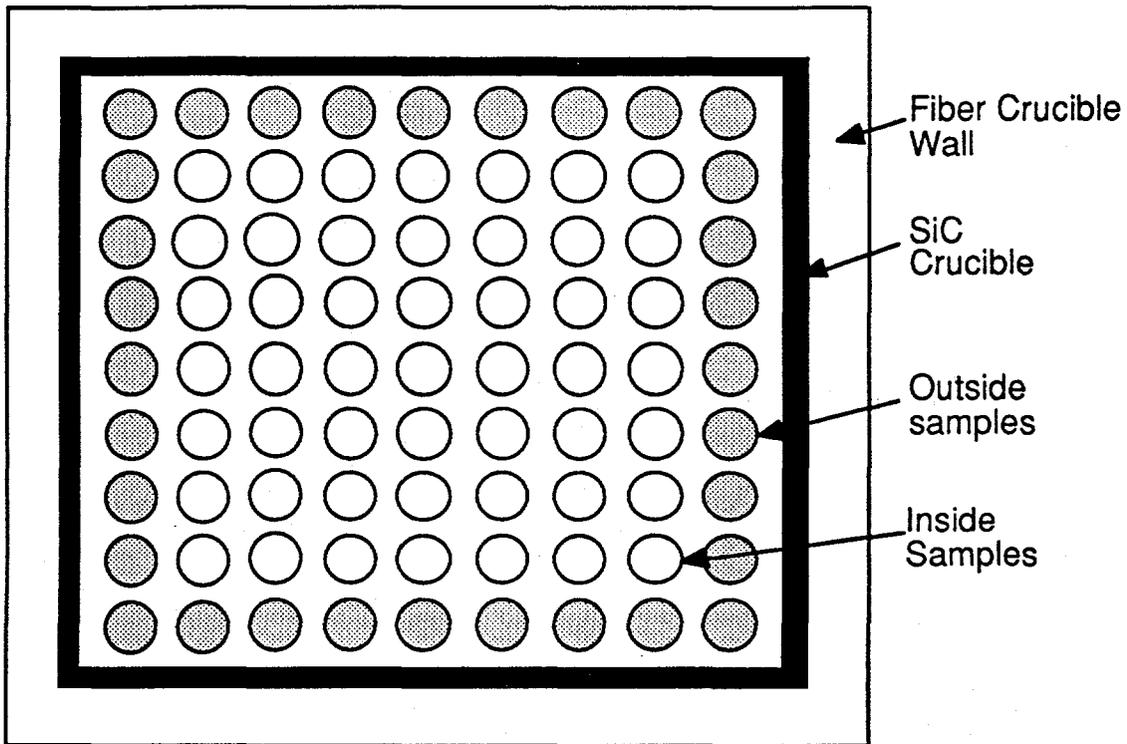


Fig. 8. Drawing of microwave package for clevis pin - crucible tests.

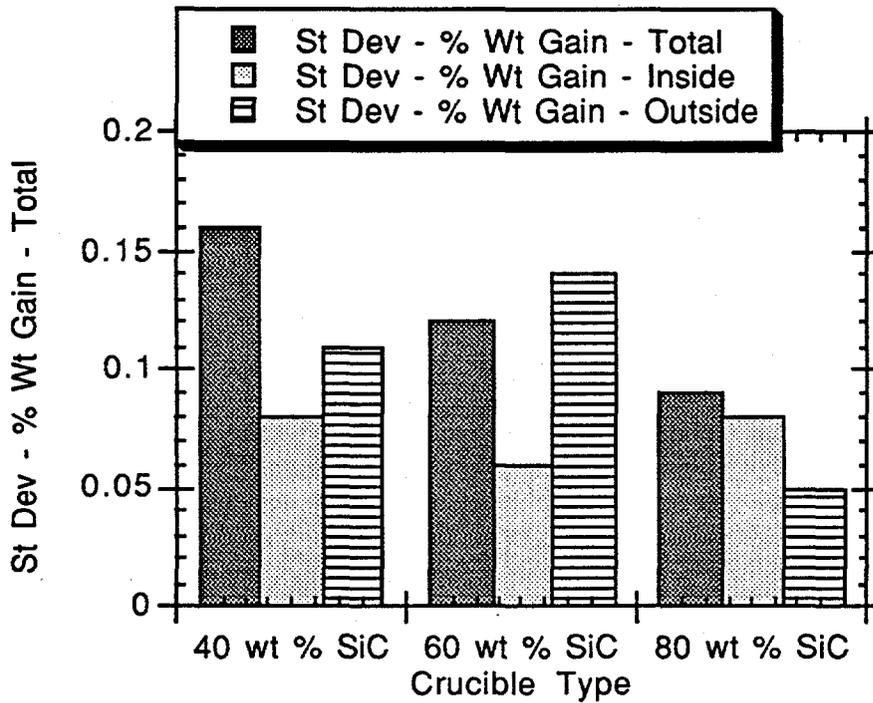


Fig. 9. Standard deviations of weight gains of the clevis pins in tests with three microwave crucibles.

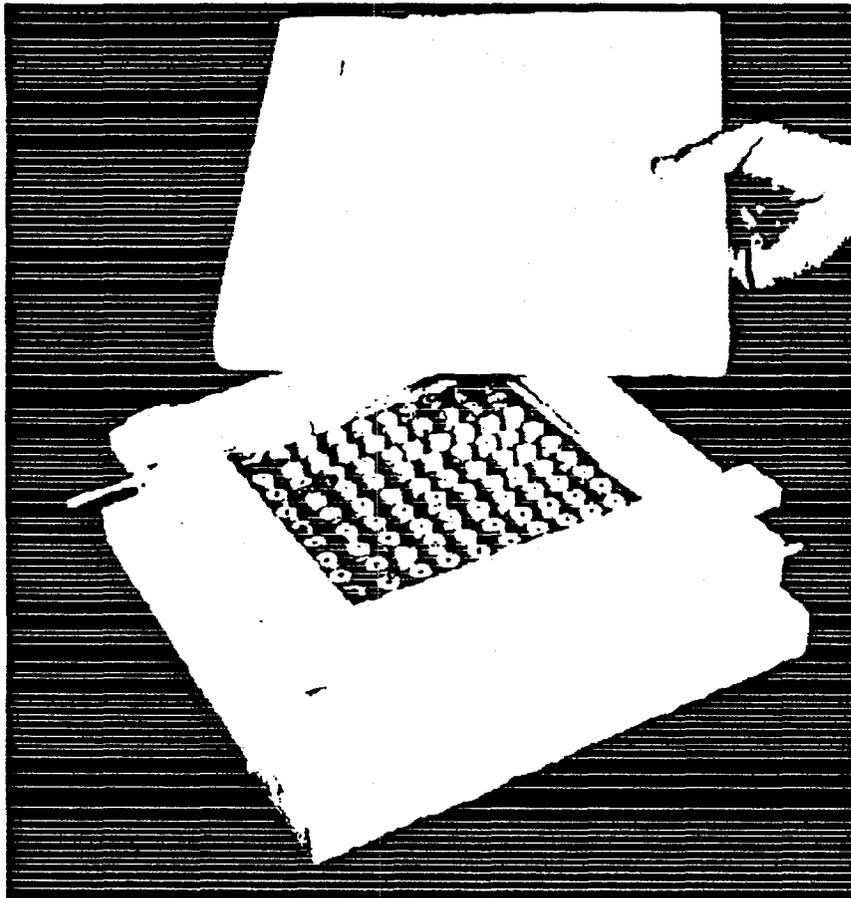


Fig. 10. Photo of 90 clevis pins nitrided using microwave heating in a 80 wt.% SiC crucible.

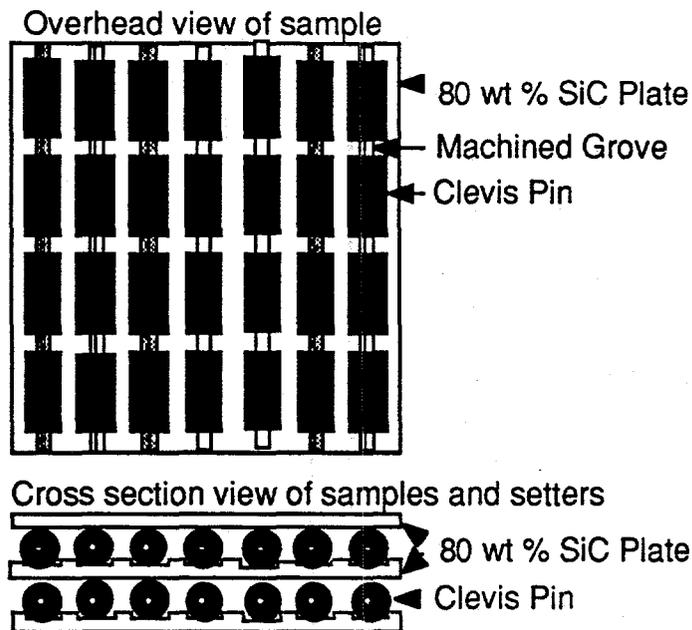


Fig. 11. Microwave crucible used for convenient sample handling during binder burnout and microwave nitridation of clevis pins.

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