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TECHNICAL NOTE

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RESISTANCE HEATED FURNACES FOR REACTION-FORMED SILICON CARBIDE ARTICLES

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The steadily increasing demand for small parts made from reaction bonded or "siliconized" silicon carbide at last is rapidly pushing manufacture of these parts out of the laboratory and pilot plant and into full scale production. New products, from washerless faucets to the most sophisticated hot gas heat exchangers are now being delivered on pre-planned schedules. Most of these products, including shaft seals and combustion chambers, have required extensive qualification testing before acceptance and many more products are in the testing pipeline, thus promising further production increases.

The resistance of reaction bonded silicon carbide to extremely aggressive atmospheres recommends this material for a number of additional applications and therefore the use of the material can be expected to increase as new applications are uncovered and as production costs are lowered.

Manufacturers of articles made from reaction bonded silicon carbide have therefore taken the big step from the realm of the scientist and process engineer into the equally demanding world of the production engineer whose charge is timely, low cost delivery of products with consistent characteristics. As a result, the "siliconizing" heat processing step has come under close scrutiny which has recently resulted in a change to resistance heated furnaces from the induction heated furnaces commonly used in the past, both in production and in the laboratory.

Much of the early work was performed in vertically oriented, induction heated, closed batch furnaces similar in concept to the design shown schematically in Figure 1. Work pieces were placed on trays installed within the heavy graphite susceptor. The susceptor was insulated by graphite powder until graphite felt became available, and was heated by a large induction coil in which the turns are electrically insulated from each other. The entire assembly was installed within a water cooled vacuum vessel. The parts themselves are heated by radiation and conduction from the susceptor, the same heat transfer mechanism that is used resistance furnaces. The closed vacuum vessel allows the initial air to be exhausted, the part to be thoroughly cleaned by out-gassing before the silicon is melted, the chamber to be back filled with pure inert gas and the infiltration step to be performed under closely controlled conditions. Pressure changes can be used to assist the infiltration process as required.

The initial process was performed in this type of induction heated furnaces. It was assumed that resistance furnaces would not be suitable because the element would be exposed to the silicon vapor and would then be rapidly converted to silicon carbide, thus changing the resistance and requiring frequent replacement. As production needs increased so also did furnace sizes. At least one induction furnace with a volume of over 50 cubic feet has been in use for several years.

From the standpoint of the production engineer, the primary difficulty with the induction heated furnances lies in the long cycle time. Many of the earlier furnances were made with relatively small power supplies which limited the heating rate. Production experience has shown that rather fast heating rates are tolerated by the green parts and the size of the induction power supplies has grown in later models with a resulting steep increase in the first cost of the equipment.

The time to cool the work for removal after the infiltration and reaction steps are completed has also been very slow in the induction heated furnaces. One of the reasons is the very heavy insulation required to protect the coil from the heat of the susceptor. The heavy insulation also slows the removal of heat when it is desired to cool the work pieces. The use of recirculated cooling gas has been attempted to assist in reducing the cooling time. The hot zone and susceptor bungs are opened at the top and bottom of the work zone and a blower installed in the chamber recirculates the inert gas over the work within the susceptor and in a return pak along the cold chamber walls which act as a heat exchanger. Such systems have been quite effective in improving the cool down time in hard metal sintering furnaces because they can be turned on at about 1200^{o}c when the natural rate of cooling is very slow. In the case of the vertical furnace, however, the effectiveness of this system is partially defeated by the horizontal work trays which limit the flow of gas. As a result, the very best cooling time is of the order of 12 hours and in the case of large furnaces, such as the 50 cubic foot unit mentioned earlier, more than a full day is required for the cool down period.

Figure 2 is a head-on view of a resistance heated horizontal furnace used in production for this purpose. The work is placed in rectangular graphite boxes outside the furnace and the boxes are then loaded into the furnace by means of a fork lift truck. Doors are provided at both ends of the furnace to simplfy handling the work boxes and to assure complete access for maintenance. The work boxes are heated by radiation from the tubular graphite resistance elements, arranged into an element assembly. Insulation is provided by graphite felt and occasionally graphite board is added for increased resistance to the silicon vapor which escapes from the work boxes. The felt is attached to a metal retainer which supports it and the entire assembly is also installed in a fully water jacketed chamber. The entire system then takes the form of the well-proved cold wall vacuum furnace.

Figure 3 illustrates an even larger production furnace of 25 cubic foot capacity. Since some of the work piece designs allow the pieces to be cooled rapidly, this model was equipped with a gas quench assembly. The unit illustrated can be used to remove heat at the rate of 2,000,000 BTU per hour and thus shorten the whole cycle drastically, provided the work pieces can withstand the thermal shock. In fact, the entire system is designed to process a load of 1100 pounds of fixtures and parts in a floor to floor time of eight hours. Some parts are indeed being processed by the furnace user in this time while other parts require a maximum of twelve hours floor to floor time.

One of the operating concerns in any siliconizing furnace is the probability of accidents which result in spilling molten silicon. The lower elements are vulnerable to such spills. Changing one or more of the tubular elements illustrated in Figure 2 can be done without removing the entire assembly, even when the tubes are partially converted to silicon carbide. Most repairs can be made with only an hour or two down time because the individual elements can be removed and replaced without removing the entire element assembly.

In order to eliminate down time for this reason, the furnace shown in Figure 3 was made without bottom elements (as shown in Figure 4). The heating element assemblies in this case are suspended on each side of the work zone. With this design, which is usually used only on very small furnaces, temperature uniformity becomes an immediate concern. However, the corners and center of this furnace were measured in a MIL SPEC. uniformity test and found to be within the plus/minus 10^{o}c specification for the furnace. Thus, the user is assured that each piece part in a full load will arrive at the prescribed reaction temperature within this tolerance. Consistent, high-yield production can therefore be expected.

When specifying a furnace for the reaction bonding operation, the production engineer has a number of factors to consider and may not consider a simple scale-up of the original laboratory development equipment to be the most efficient production means. Recognizing that the work pieces do not care whether they are heated by radiation from resistance elements or by radiation from an induction heated susceptor, furnace selection considerations include such matters as convenience of operation, first cost, production time lost to maintenance and cooling time. The horizontal furnace is generally more convenient to load, unload and to service. Further, when one compares the number of small size work pieces that can be placed in a rectangular furnace hot zone compared to the number that can be placed in an equal volume vertical cylindrical hot zone, it is seen that approximately 15% more pieces will fit in the horizontal furnace.

A comparison of the first cost of similar size vertical induction-heated and horizontal resistance-heated furnaces offered by the same manufacturer shows the resistance heated-furnace price to be about 13% less than the induction heated model of equal size.

Similarily, when cool down times are compared, the horizontal furnace allows for more efficient use of a gas recirculation work cooling system. The work trays do not obstruct the flow of the cooling gas in a horizontal model. As a result, horizontal furnaces with a full load may be cooled in as little as two hours in some extreme cases and in six to eight hours in the average case. This compares to the cool down time of the vertical furnace which is usually on the order of twelve hours.

Contrary to the earliest judgements, the maintenance costs and down times have not proved to be excessive in the resistance heated furnaces. One user reports an average of 110 cycles before the felt insulation is replaced. Such element replacement as may seem desirable or necessary is accomplished at the same time. However, the upper elements, which are not subject to falling debris and molten silicon, operate over 200 complete cycles before requiring replacement. As new materials which are more resistant to silicon vapor become available, the time between scheduled maintenance work will increase.

To conclude, the manufacture of infiltrated reaction-bonded silicon carbide articles has now moved from the laboratory and the pilot plant to full scale production. As additional furnace capacity is required, resistance heated furnaces have been selected for reasons of cost and efficiency and they are now demonstrating faster cycles and more consistent results than the earlier induction heated furnaces. Scheduled production of these advanced ceramic articles has now become a fact of life.

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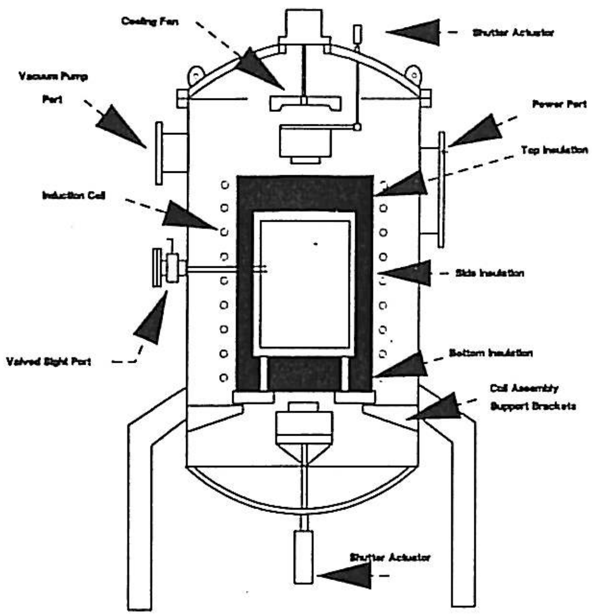

Figure 1
Vertical Induction Heated Batch Furnace

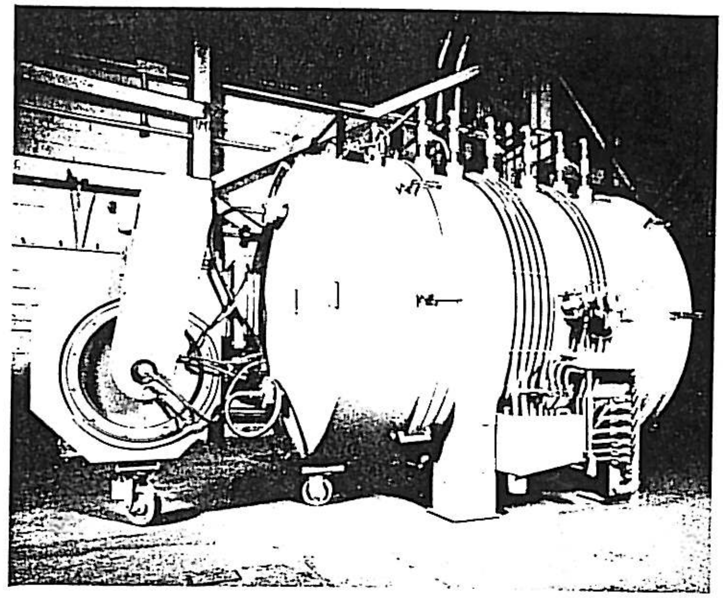

Figure 3
Resistance Heated Furnace with Gas Quench

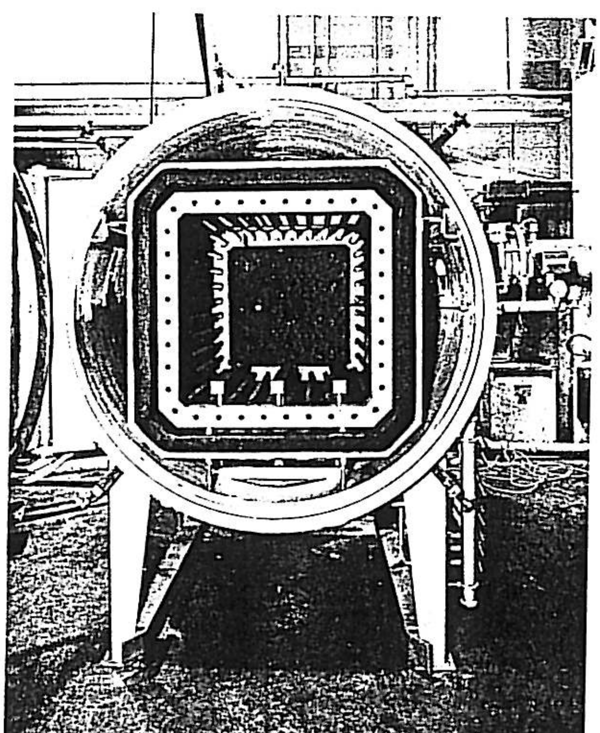

Figure 2
Resistance Heated Horizontal Furnace

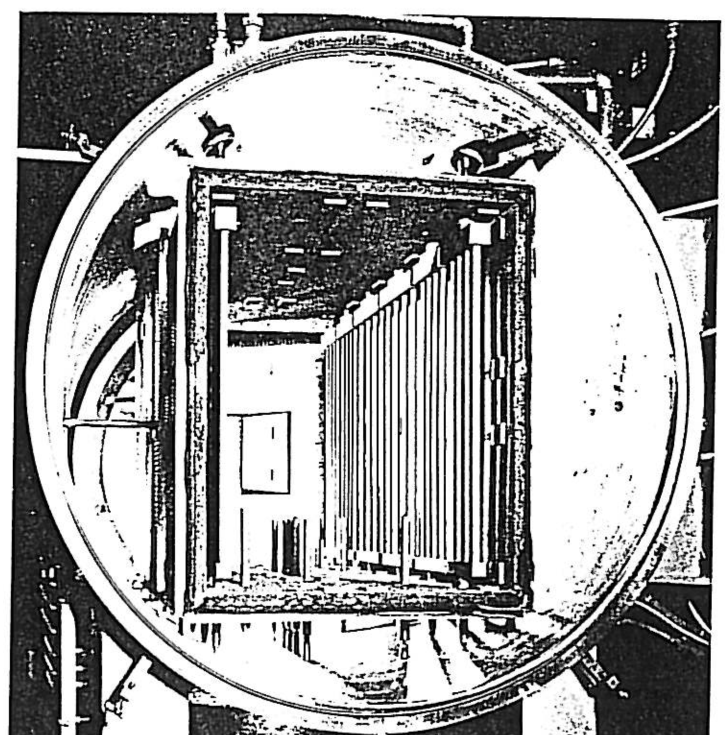

Figure 4
Resistance Heated Furnace with side elements only.