

NOTES ON THE USE OF FUSIBLE TEMPERATURE INDICATORS
TO BOUND THE TEMPERATURE OF HOT GRAPHITE
IN THE NAVSWC HYPERVELOCITY WIND TUNNEL FACILITY

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Summary. Temperature sensors, which indicate by melting whether or not a particular temperature level was reached, were used to determine upper and lower bounds on the peak operating temperatures of a clamping fixture that is made of graphite. The clamp secures an electrically powered carbon-carbon heating element to its graphite support base in a pressure vessel that is part of the Hypervelocity Wind Tunnel located at the Naval Surface Warfare Center in Silver Spring, Maryland. During wind tunnel testing the clamp is exposed to an extremely hostile environment that includes: temperatures to 3700°C <6700°F>, intense thermal radiation, pressure to 22,500 psi, hot chemically reactive carbon and carbon vapor, electrified parts, stray electromagnetic fields, and finally, an intense aerodynamic loading.

I. INTRODUCTION

The Hypervelocity Wind Tunnel (Tunnel 9) is located at the Naval Surface Warfare Center in Silver Spring, Maryland. A schematic of the Tunnel 9 Facility is shown in Figure 1. Tunnel 9 is a "blow-down" tunnel that runs at a test Mach number of either 8, 10, or 14. During a tunnel run, a constant hypersonic flow condition can be maintained in the test section for approximately 1/3 to 15 seconds depending on the particular test condition being run.

Tunnel Components

Figure 1 depicts the major components of the Hypervelocity Wind Tunnel. A "Gas Heater Vessel" delivers heated nitrogen gas to the test section during wind tunnel operation. Three "Gas Driver Vessels" supply nitrogen gas to the gas heater vessel (the three driver vessels are manifolded together and act as one large high-pressure gas source). "Pressure Control Valves" between the driver and heater vessels are used to regulate the gas pressure in the heater vessel during a tunnel run by controlling the amount of driver gas admitted into the heater vessel. The "Diaphragm Section" contains the wind tunnel nozzle throat and metal burst diaphragms used to start the tunnel. The 40 foot nozzle accelerates the heated tunnel gas to hypersonic velocities. The "Test Cell" contains the wind tunnel model. A 150 foot long diffuser pipe connects the test cell to the "Vacuum Sphere".

Tunnel Operation

In preparation for making a tunnel run, the driver vessels and heater vessel shown in Figure 1 are each filled with a fixed quantity of nitrogen gas. This gas is then heated inside the vessels by electrically powered heating elements. About 15 minutes is the time required to heat the gas in the heater vessel. It is necessary to pre-heat the gas

in the heater vessel to either 1650°C <3000°F> (for a Mach 14 run) or 815°C <1500°F> (for Mach 8 or 10) in order to prevent condensation of this nitrogen in the test-section of the tunnel during the wind tunnel run. The final gas pressure in the heater at the end of the heating process may be as high as 22,500 psi. In addition, before running the tunnel, the air is evacuated from the vacuum sphere.

A wind tunnel blow is initiated immediately after the heating process by bursting a set of metal sealing diaphragms that are located immediately upstream of the nozzle. The rupturing of the diaphragms releases the hot, high pressure nitrogen from the heater vessel into the nozzle. As the gas expands through the nozzle towards the test section it accelerates to a hypersonic velocity and enters the test section at the required velocity, pressure, and temperature. After flowing past the test model, the gas exits the test section and enters the diffuser pipe which carries the exhaust gas into the large vacuum sphere. During the tunnel run, driver vessel gas, supplied to the heater vessel at a controlled rate, acts as a fluid piston that pushes the hot gas out of the heater vessel and through the nozzle throat at a constant pressure and temperature. By controlling the flow of driver gas into the heater, constant flow conditions are maintained in the test section during the tunnel run. Immediately after the hot gas in the heater is expended, the pressure control valves between the driver and heater vessel are closed and the remaining nitrogen in the heater vessel is vented out through the nozzle. Depressurization rates of up to 3000 atm/sec may occur during the venting process.

II. BACKGROUND

Description of Gas Heating System and Requirement for Temperature Measurements

The heating element, shown in Figures 2 and 3, is secured to the support base with six graphite setscrews (three per side) which clamp the element's two legs to two mating tongues on the support base as shown in Figure 3. The setscrews force the ends of the two legs of the element against the tongues of the support base and thereby produce sufficient pressure at the connection to maintain adequate mechanical and electrical contact between the element and base and thereby prevent local heating and electrical arcing at the connection. The graphite heating element is powered by a 60 hertz AC, single phase, source operated at an average current of 5500 amps.

The graphite heating elements sometimes fail, by fracturing, after being in service for only a brief time. Replacement after each element failure is costly and time consuming. To increase the

reliability of the heating elements a carbon-carbon heating element was developed to replace the weaker graphite elements. The carbon-carbon heating element required a better clamping method because the old setscrew clamp, when used with the carbon-carbon element, loosened during every wind tunnel run. The loosening of the electrical connection can lead to overheating and arcing and subsequent damage to the heating element and support base and loss of the run. The amount of torque that can be applied to the setscrews is limited by the bending strength of the tongue portions of the support bases. A relatively low force is all that is required to break off the tongues. Therefore, the setscrews can supply only a relatively small clamping force which is marginally adequate and, therefore, even a small loss in the clamping force can be detrimental to the integrity of the joint. It was not desirable to open the heater vessel and tighten the setscrews after each run because this time consuming operation would defeat the time and cost savings achieved by using the carbon-carbon heating element.

An alternate clamping arrangement, in which the element's legs are bolted directly to the tongues as shown in Figure 4, is not limited by the bending strength of the support tongues, consequently, a much greater clamping force can be applied to the connection. Because the new clamp will develop a higher initial preload, it should be able to sustain a loss of a portion of its clamping force and still supply sufficient clamping pressure to the connection. The bolt fasteners for the new clamp will be loaded in tension (unlike the setscrews in the old clamp design which are loaded in compression) and will be subjected to an elevated temperature. Because of the anticipated tensile loading of the bolts, it was decided not to fabricate the bolts out of bulk graphite because, due to its brittle nature, bulk graphite has a low tensile strength. Therefore, knowledge of the operating temperature of the joint was needed to select a material to be used for the tension bolts and washers for the new design and to size the fastener hardware to compensate for the different coefficients of thermal expansion of the bolt and heating element materials.

Measurement of the Temperature of the Element-Base Connection

The development of a connection to reliably clamp the carbon-carbon element required knowing an upper bound for the peak operating temperature of the connection. It was necessary only to bound the temperature at the outer diameter of the heating element where it contacts the center setscrews because an analysis indicated that this location should be the hottest part of the connection. Additionally, it was necessary to bound the temperature of the collar portion of the support base where the center setscrew ports are located because the bolts and washers would be affected by the heating of the collar.

Selection of the Measurement Technique

Several schemes to accomplish the temperature measurements were proposed. The most common methods for high temperature measurements, i.e., thermocouples, radiation thermometry, and commercially available fusible temperature indicating paints (melting paints) were all considered in the initial evaluation of available methods.

The area in which the measurements were required was characterized by hostile conditions. The heating element itself reaches a peak operating temperature of perhaps 2750°C <5000°F> and emits intense thermal radiation. The gas pressure may reach as high as 22,500 psi and depressurization

rates of up to 3000 atm/sec may be experienced during a wind tunnel run. During the heating period a 60 hertz alternating current, single phase, that averages 5500 to 6500 amperes, is passed through the support base and heating element. The electrified parts and electromagnetic interference from the electrical power are potential sources of error for electronically based measurement techniques.

The use of a radiation thermometry apparatus to obtain the temperature measurements was declined due to the complexities involved in mounting such a device inside the pressure vessel in the high temperature environment. Furthermore, the temperature data were required in internal locations that were not readily viewable by optical means. A tungsten-rhenium thermocouple device was considered but was not selected because questions arose concerning the ability of the electrical insulator materials to provide adequate insulation and shielding of the thermocouple wires at the elevated temperatures anticipated and in the carbon atmosphere (the ceramic insulators such as boron nitride and alumina, become electrically conductive at high temperatures or if sufficient carbon diffuses into these materials). Under high temperature conditions (starting at approximately 1400°C <2550°F> for magnesia insulated tungsten-rhenium), the electrical shunting effect caused by the lowered resistance can begin to introduce an observable error in the measurement. Under ideal conditions this shunting error can be calibrated out, however, it was believed that it would be a non-trivial matter to obtain a calibration environment which adequately modeled the actual test conditions.

Fusible temperature sensors indicate, by melting, whether or not a particular temperature level (i.e. the melting point of the sample) was reached. If a particular sample does not melt then its rated temperature represents an upper bound of the temperature level reached. Conversely, if a particular sample melts then its rated temperature represented a lower bound of the temperature level reached.

The fusible indicators have several advantages that made them ideal for making the required measurements. For example, the temperature at which the indicators melt is inherently unaffected by temperature and, as will be shown later, the melting temperature is little affected by ambient pressure. The indicators can be used in hard-to-access locations because they are not bulky and require no lead wires. They are unaffected by low frequency electromagnetic energy and non-conductive indicators should not be affected by electrified heater parts. Finally, with proper selection of the indicators, an extremely wide temperature range appeared feasible.

For the reasons stated, the fusible temperature indicator method was selected to carry out the measurements. The technique described here employed metallic and commercial paint samples.

III. BACKGROUND ON ACCURACY OF FUSIBLE TEMPERATURE INDICATORS

Effect of High Pressure on Melting Point

The melting points of solid materials are, in general, weak functions of pressure -- at least for the range of pressures that exists in the heater vessel of Tunnel 9 (0 to 22,500 psi). The effect of pressure, p , on the melting temperature of a pure substance in degrees absolute, T_m , can be expressed by the Clausius-Clapeyron equation²:

$$\frac{dT_m}{dp} = T_m \frac{(V_L - V_S)}{\Delta H}$$

where V_L and V_S are the molar volumes of the liquid and solid, respectively, and ΔH is the molar heat of fusion. For most substances the melting point increases with increasing pressure because most substances experience an increase in volume upon melting (a notable exception is water). The variation of melting point with pressure for elements and various compounds can be readily obtained from the experimentally derived phase diagrams that are available for these materials^{3,4}. Specific data for the melting points was not available for the high pressures of interest here, therefore, the trends discussed below, for the materials for which data were available, were taken as indicative of the high pressure effect on melting of the commercial paints. Figure 5 shows examples of phase diagrams for iron, copper, and aluminum⁵. The melting curves in Figure 5 indicate that the melting points for these elements initially rise with pressure. Table 1 shows the initial slopes (the change in T_m with pressure) for iron, copper, and some other common materials^{3,4}. The initial slopes are +0.32°C per 1000 psi for copper and only +0.24°C per 1000 psi for iron. The variation in melting point, for the materials listed in Table 1, is small for the range of pressures experienced in the Tunnel 9 heater vessel. For example, the increase in T_m in going from atmospheric pressure to 22,500 psi is only +7.2°C for copper and +5.4°C for iron. In summary, the pressure effect on melting point was taken to be insignificant for the range of pressures that exist in the heater vessel of Tunnel 9.

Eutectic Melting Effect

A potential source of error when using the fusible indicators to measure temperature is eutectic melting⁶. When two different solids are placed in intimate contact or alloyed together, eutectic melting is the partial or complete liquefaction of the contacting materials at a temperature that is below the melting point of either of the two pure components. Eutectic melting is commonly observed when salt (calcium chloride, CaCl_2) is spread on an icy walkway in wintertime. Application of the salt to the ice results in the formation of brine which has a lower melting point ($T_m = -51^\circ\text{C}$) than either the pure ice ($T_m = 0.0^\circ\text{C}$) or the pure salt ($T_m = 772^\circ\text{C}$ for CaCl_2). The eutectic melting for the $\text{H}_2\text{O}/\text{CaCl}_2$ system is depicted on the phase diagram shown in Figure 6⁵. The phase diagram shows that some liquid phase will be observed at or above -51°C for a large range of component proportions. Only at the eutectic composition (which for H_2O and CaCl_2 is approximately a 71/29% ratio by weight) does the solid solution exhibit a distinct melting point at -51°C . The other compositions begin melting at -51°C but, as the phase diagram shows, melting is not complete until some higher temperature (which depends on the composition) is reached. A phase diagram for the rhenium-carbon system in Figure 7 shows a eutectic melting point of 2480°C . By contrast the melting point of pure rhenium is 3180°C and pure carbon sublimates at 3704°C . Similarly, pure iron melts at 1530°C but the melting point is depressed to a eutectic at 1125°C when alloyed with carbon⁸. For some systems the effect is small, for example, the copper-carbon system exhibits a eutectic temperature which is less than 1°C below the melting point of pure copper. The examples of eutectic melting given above show that this phenomenon can significantly depress the melting temperature of a solid. The existence of the eutectic melting phenomenon makes it important to

From the Greek "eutektos" which means "easily melted"

know how a fusible temperature indicator will respond in this context when contacted by the materials in the environment in which the indicator will be used (such as the crucible material in which the fusible indicator will be held).

Effect of Chemistry

The occurrence of an adverse chemical reaction can render a fusible indicator ineffective as a temperature indicator. A variety of reactions is possible depending on the materials employed. Specifically, for the Tunnel 9 heater environment, the potential reactions of the sample or crucible with graphite, carbon vapor, and nitrogen must be known because these elements are present in the high temperature areas of the heating system. Some specific reactions with carbon include the formation of carbides or reduction of oxides by the carbon. A fusible sample may form a stable carbide or nitride layer on its surface at a temperature that is below the rated melting temperature of the sample and the carbide or nitride layer may not melt at the sample's rated temperature. Melting of the interior of the sample may occur, however, the un-melted carbide or nitride outer layer may cause the sample to retain its original shape even though the interior material has melted. If the sample retains its original shape beyond the rated melting temperature, it may be impossible to determine by visual inspection whether or not the melting has occurred and hence whether or not the melting temperature was reached. This phenomenon was observed by the author during a calibration test that was being conducted with a sample of an alloy of aluminum being heated in air. The melting point of the aluminum sample was reached, however, only the interior metal melted while, on the surface of the sample, a skin formed, which did not melt and thus contained the molten interior metal until it re-solidified in a form that more or less resembled the original shape of the sample (the sample was a plate about 2 inches square by 1/8 inch thick). Apparently, an un-melted natural oxide coating had formed on the sample's surface that was strong enough to contain the molten aluminum in the interior of the sample. This suggested that for small samples, the natural oxide, by maintaining the original structural shape of the sample beyond its rated temperature, may prevent a visible indication that the melting temperature was reached.

IV. FUSIBLE TEMPERATURE INDICATOR USE IN TUNNEL 9

Initial tests with Commercial Fusible Paints Applied to Graphite

The "Omegalag" paints (sold by Omega Engineering, Inc.) were first tried on the graphite heater parts. The temperature ratings of the paints used ranged from 121°C to 1371°C in 121°C increments (< 250°F to 2500°F in 250°F increments). Selected paints were painted directly on the outer surface of the graphite support base. Also, graphite setscrews were fabricated which had a small flat machined on one side on which lines of paint were applied. These screws were then used to clamp the heating element during a heating cycle and then removed and the paints inspected to obtain temperature indication data.

Early in the test program serious problems arose when these paints were used directly on the graphite parts and also when the paints were exposed to the carbon vapor which is present in the gas inside the heater vessel during the heating process. If they were checked after being subjected to only one heating cycle, the 1093°C and 1371°C paints could usually be interpreted easily because they were always found in the original unmelted form,

presumably because the temperature usually stayed sufficiently below the rated temperature of these paints. If more than one cycle was placed on the 1093 or 1371 paints they would often become too obscured with carbon dust contamination which made the paints unreadable. The paints below the 1093 range were beset by unusual reactions after being subjected to only one heat cycle, such as complete disappearance of a paint, complete transformation to a black carbon-dust like appearance, and one case where the 816°C <1500°F> paint was apparently reduced by the carbon to a metal or metal carbide which appeared as small metal-like spherical beads which were bonded to the graphite. It was determined, for some cases, that the operating temperature range was between 260 and 1093°C <500 to 2000°F>. However it was desired to narrow this temperature range more. In particular, it was desired to show that the temperature did not exceed 700°C because an inexpensive nickel-based superalloy such as INCONEL X-750 or 718, which maintains high strength up to 700°C, could then be used as bolt and washer material for the clamp. Unfortunately, the 538, 677, and 816°C <1000, 1250 and 1500°F> paints never yielded any useful temperature data to help narrow the operating temperature range because these paints could not be made to perform properly.

The paints were checked by bench testing them on stainless steel and graphite substrates. Painted samples were simply back-heated with a butane torch and in all cases it was very easy to see exactly when a paint melted because the paint would liquify and form a glossy beaded appearance. The resolidified portion of a paint was readily determined because the resolidified paint possessed a glossy or crystalline solid surface which contrasted with the matte powdered appearance of the unmelted paint. The fusible temperature paints were originally designed to be used on metals for welding operations (for example to indicate when a preheat temperature is reached) in oxidizing or inert atmospheres. Because the paints work well on metals it was decided that, for the Tunnel 9 tests, the paints would be held in a stainless steel crucible. It was determined that for the paints to work reliably they also should be protected from contact with graphite and carbon vapor and from contact with electrified parts, and therefore, the paints and metal crucible should be housed in a chemically inert and electrically insulating jacket. Selected metallic materials were also utilized as melting samples, in addition to the paints, as an alternate source of data. Boron Nitride (BN) refractory material, which was readily available, was used as both an inert protective jacket and as a crucible for the metal samples.

Nature of Boron Nitride

Hot pressed boron nitride (BN) is a relatively inert, non-toxic refractory material. BN is a white solid and has a crystal structure and density nearly identical to that of polycrystalline graphite. BN possesses good thermal conductivity but, unlike graphite, is an excellent electrical insulator. High purity grades such as Union Carbide grade HBC or Carborundum grade AX05 can be used in inert atmospheres at temperatures up to 3000°C <5400°F>. The material can be easily machined (dry) using conventional machine tools although the dust from machining must be controlled because it is an eye and respiratory irritant. BN crucibles are commonly used to contain many molten materials often at temperatures far above their melting points⁸. In particular, BN can safely contain iron, copper, and aluminum up to their respective melting temperatures while exhibiting no eutectic melting. The BN material possesses structural and thermal properties similar to those of graphite. Boron nitride will begin to react with graphite only at temperatures above approximately 2200°C <4000°F> where it forms

boron carbide (B₄C) a shiny very hard refractory. BN will react slowly with molten stainless steel (type 304) but even so, according to one BN manufacturer, no eutectic melting will be observed between BN and the steel up to the melting point of the steel (1400-1450°C). BN is not recommended for use with molten beryllium, nickel, platinum, uranium, and the oxides of lead, antimony, bismuth and copper⁸.

V. A TEMPERATURE INDICATING SETSCREW AND PLUG FOR USE IN THE HOSTILE TUNNEL 9 HEATER VESSEL ENVIRONMENT

Setscrow Concept

The unique properties of boron nitride described above were utilized to develop a temperature indicating setscrew and a temperature indicating plug insert that would function in the hostile environment of the Tunnel 9 heater vessel. Figures 8 and 9 show the concept for the temperature indicating setscrew used in the graphite support base for the Tunnel 9 heating element. The setscrew device was used to bound the temperature of the collar portion of the graphite support base at the setscrew port locations. The temperature indicating setscrew, which in operation simply replaced a graphite setscrew in the heater support base, consisted of 5 principal parts: a BN setscrew body, a (type 304) stainless steel boat that held the temperature paints in two V-groove troughs, a BN retainer-seal plug, a BN seal cap, and a tap-hole ballast plug. All internal voids in the screw were vented to the outside in order to avoid a large pressure differential across the device when pressurizing and depressurizing the nitrogen gas in the heater vessel. The core of the BN body was threaded with a 1/4-20UNC internal screw thread which held the male-threaded boat and sealing plug in a fixed position in the body and also provided adequate venting of the tapped hole and internal voids formed by the troughs in the stainless boat. The ballast plug filled the small unthreaded void which was left at the bottom of the threaded hole by the tap-drilling operation during fabrication of the body. A reservoir was provided in the head of the BN screw body for the purpose of catching and holding any molten steel that might leak past the threaded retainer plug should the temperature become high enough to melt the stainless boat.

A medicine dropper was used to fill the stainless boat with the temperature paints. The paints, on drying, formed a rigid crust on the exposed surface that firmly held the paints in the groove. After tightening the setscrew in position in the heater support base, the steel boat would have to be rotated so the troughs holding the paints were facing upwards to ensure that the paints, if melted, would not drip out and solidify in the threads and thus freeze the boat inside the BN body. After positioning the boat upright, the retainer plug was threaded into the BN housing and tightened against the steel boat to help hold the threaded boat tight against the screw threads in the BN body. This helped to maintain intimate thermal contact between the boat and the BN body. Finally, after positioning the internal setscrew parts, the vented seal cap was threaded onto the head of the setscrew by hand. This cap contained a small central vent hole that allowed nitrogen to vent into or out of the setscrew body.

Plug Concept

The setscrew described in the previous section indicated the temperature of the collar portion of the heater support base where the center setscrew ports are located. An upper bound on the temperature at the outer diameter of the heating element, where it contacts the center setscrews, was also desired as mentioned earlier. To accomplish this, two

temperature indicating plugs, shown in Figure 10, were used. One plug body was made of boron nitride and carried a cylindrical stainless steel boat which, in turn, carried the temperature paint in small holes in the ends of the boat. The other temperature indicating plug held three small metallic ring shaped melting samples which were sandwiched between BN sealing discs. The sealing discs, which were press fitted into the plug body, were designed to contain the melted samples. The end faces of the plug body were grooved and the sealing discs each had a center hole and grooves to allow venting of nitrogen into and out of the assembled device. The metal ring samples were made of either stainless steel, copper, brass, or an aluminum alloy. Table 2 gives the atmospheric melting and boiling points of these metals. The rings were sized so if one melted it would fill less than half the space between the seal discs and thus would not leak out of the center vent holes in the discs.

Operation of Setscrew and Plug

In operation, a setscrew and plug were assembled as shown in Figure 9. First, a BN plug was inserted into the setscrew port then a pyrolytic graphite (PG) spacer placed in the port behind the BN plug, and the BN temperature indicating setscrew was then threaded into the port and tightened against the PG spacer with a torque of 10.0 inch-lb applied to the setscrew. The PG spacer, which has a very low thermal conductivity across its thickness, acted as a thermal insulator to minimize heat transfer between the plug and setscrew (the PG spacers have a thermal conductivity in the thickness direction of only 0.002 cal/cm-sec-°C at 1650°C⁹). The BN plug was thus thermally insulated from the setscrew by the PG spacer and, because the BN plug was in intimate thermal contact against the outer diameter of the heating element, the plug presumably would sense the temperature at this location as desired.

VI. EXPERIMENTAL RESULTS OF THE USE OF TEMPERATURE INDICATING SETSCREWS AND PLUGS IN THE TUNNEL 9 HEATING SYSTEM

Wind Tunnel Run #1 (High Pressure)

The first wind tunnel run was made using the highest standard gas pressure of 22,500 psi. A BN setscrew assembly containing temperature indicating paints and a BN plug assembly containing metallic ring samples were installed as shown in Figure 9 in one of the center setscrew ports in the Mach 14 heater support base before making the tunnel run. The BN setscrew held a 677°C (1250°F) paint and a 954°C (1750°F) paint. The BN plug held three metal fusible ring samples made of alloys of copper, aluminum, and stainless steel. The alloy types and melting ranges were as follows: a 99.9% copper alloy (any of these alloys: C10200, C10400, C10500, C10700, C11000, C11300, C11400, C11600), $T_m = 1065$ 1082°C¹⁰; a 95.85% aluminum alloy (A96061), $T_m = 582$ 648°C¹⁰; stainless steel (S30400 i.e. type 304), $T_m = 1398$ 1454°C¹⁰. This first tunnel run was a standard high pressure run made with a gas pressure of 22,500 psi and gas temperature of 1650°C <3000°F>. The nitrogen in the heater vessel was heated by the carbon-carbon heating element which was operated at an average AC current of 6500 amps. The available tunnel performance data for this run indicated that a normal heating cycle was made.

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- * Due to test scheduling pressures there was not time to obtain laboratory-pure metal samples for these tests.
 - ** Unified Numbering System alloy designations are used.

After the wind tunnel blow was made the BN temperature indicators were removed and the melting samples were checked. All the external surfaces and some internal surfaces of the BN parts that were exposed to the nitrogen gas were covered with a gray/black coating (apparently of graphite) as shown in Figure 11. This coating was not a "dusting" but rather was diffused into the BN surface. A simple check of the surface resistance was performed on one of the blackened BN parts and, for comparison, on a virgin piece of identical BN material (Union Carbide grade HBC). Resistance meter probes were held on the surface of each piece with the probe tips separated by approximately 1/8 inch. Care was taken to ensure that the meter operator did not touch his hands together while holding the separate probes to prevent erroneous readings resulting from measuring the resistance of the operator's skin and body. The resistance of the virgin BN material always measured off-scale meaning the surface resistance was greater than 10 megohms. The blackened BN parts produced surface resistance readings as low as 6000 ohms. It is most likely that the black coating is electrically conductive graphite which has diffused into the surface of the BN.

Upon disassembly of the BN setscrew it was found that both temperature paints (677°C and 954°C) had turned a light gray color (originally the 677 paint was silver and the 954 paint was orange). Several of the paints were observed to change colors upon heating during bench tests and, according to the manufacturer, this does not affect their accuracy ($\pm 1\%$ of rating at 1 atm pressure). Neither paint had melted because both paints were found to still be in powdered form. Except for a gold tint which developed on its surface, the stainless steel boat which held the paints was unchanged.

Disassembly of the BN plug revealed that apparently none of the three metal samples had melted. The copper sample appeared shiny and like new; the surface of the stainless steel sample was still shiny but had developed a bluish tint; the aluminum sample had developed a thin dull-black coating which was easily scratched to show the metal beneath. A thicker scale was present on the inner diameter of the aluminum sample. All the BN parts were in excellent condition so these BN parts could be re-used if necessary. The disassembled setscrew and plug parts are shown in Figure 11 and the metal ring samples are shown in Figure 12.

The results given above were interpreted as follows. The BN plug (backed by a pyrolytic graphite disc) was designed to indicate the limit temperature of the base of the carbon-carbon heating element as explained earlier. The steel and copper samples in the BN plug did not melt. The aluminum sample appeared not to have melted, however, in a subsequent test (to be described below) another aluminum ring sample retained its ring shape well beyond the melting temperature of the aluminum (presumably because, for the small sample size used, the natural oxide film present on the sample's surface helped the sample retain its ring shape even though the interior metal was temporarily molten). Therefore, the performance of the aluminum led to an inconclusive result. The copper sample, which clearly did not fuse, indicated that the temperature of the base of the carbon-carbon heating element did not exceed the melting temperature of the copper which is 1082°C. The BN setscrew was designed to indicate the limit temperature of the outer collar of the graphite support base that contains the threaded setscrew ports. The 677°C paint used in the setscrew did not melt, therefore, the temperature at this center setscrew location did not exceed 677°C.

Wind Tunnel Run #2 (low pressure)

After the high pressure tunnel run, a low pressure tunnel run was made with the lowest standard heater gas pressure of 3300 psi. Two BN plugs were installed in opposite halves of the Mach 14 graphite heater support base, one in each center setscrew port as shown in Figure 9. Both plugs were backed by a PG disc and were clamped tight against the base of the heating element by either a graphite or BN setscrew as shown in Figure 9. One plug contained a stainless steel boat that held 538°C <1000°F> and 816°C <1500°F> melting paints. BN seal plugs were installed on both ends of this BN plug to help keep out carbon vapor and keep the melting paints in. The other BN plug contained three metal melting samples made of a naval brass alloy (C46400), $T_m = 887^\circ\text{C} - 899^\circ\text{C}^{10}$, an aluminum alloy, and stainless steel. A BN setscrew was installed in the graphite heater base in the same setscrew port in which the BN plug holding the three metal samples was installed.

This low pressure wind tunnel run was made with a heater gas pressure of 3300 psi and a gas temperature of 1760°C (3200°F). Again the carbon-carbon heating element, operating at an average current of 6500 amps, was used to heat the nitrogen gas in the heater vessel. Time history profiles for the heating element resistance for both this tunnel run and the previous run are shown in Figure 13. Note in Figure 13 that an anomalous abrupt rise in the heater resistance occurred about 5 minutes into the heating period. In spite of the anomaly, the gas heating process was essentially normal because the tunnel operator was able to maintain a 6500 amp current through the heating element for the entire 15 minute heating period.

After making the tunnel run the heating system was inspected. The virgin white BN parts had once again developed a blackened surface coating. It was found that the threaded tongues of the graphite base halves were both broken off at the roots. Areas were found on the fracture surfaces of the tongues where the graphite had melted and flowed and then re-solidified indicating that intense heating had occurred in these areas during the heating period (possibly due to electrical arcing). The fracture surfaces also contained areas where mechanical brittle fracture had occurred.

The fusible samples (both metallic and paint type) and the stainless steel boats used to hold the paint samples had all melted as will be discussed here. The BN seal discs had prevented the molten metal ring samples from leaking out of the BN plug that held the metal ring samples. The brass and stainless steel ring samples re-solidified inside the BN plug in the form of "B-B" size pellets about one-tenth of an inch in diameter (Figure 12) and were easily removed for inspection. The formation of the pellets upon melting made it very easy to tell that the samples had melted. The aluminum sample did not form a pellet like the brass and stainless steel samples did. Rather the aluminum sample retained its original ring shape although the shiny machined surfaces of the ring had been transformed to dull grey-black scale with most of the scale appearing on the inner diameter surface (Note: the aluminum ring was found intact after the test and was subsequently divided into three pieces, as shown in Figure 12, for inspection purposes). There is virtually no doubt that the temperature was high enough to melt the aluminum. The temperature was probably not high enough to melt the natural aluminum oxide which certainly coated the aluminum sample. As described earlier, this outer oxide coating may have held the molten aluminum in place until it re-solidified. This may explain why the aluminum sample retained its original ring shape. In addition, a non-pure alloy (type A96061) was used that contained small

amounts of other elements one of which may have reacted with the nitrogen gas in the heater vessel and formed the heat resistant scale on the sample.

The BN plug that held the stainless steel boat which carried the two paints, upon opening, revealed that the steel boat (and presumably the lower melting point paints) had melted and re-solidified inside the plug.

The BN setscrew developed a hairline crack near the tip through which some molten steel leaked and re-solidified in the threads between the setscrew and graphite base and this locked the setscrew in place which made it necessary to cut the heater support base in order to retrieve the BN setscrew. Most of the molten steel did not leak through the hairline crack and was contained by the threaded seal plug at the opposite end of the BN setscrew assembly thus preventing the steel from leaking back into the reservoir located in the head of the setscrew.

The results of this low pressure run were interpreted as follows. The stainless steel boat parts in both the setscrew and plug melted, therefore, the temperature reached or exceeded 1454°C <2650°F> at the points where the steel parts were located. Therefore, the lower bound on the temperature which was reached in these setscrew and plug locations for this run was 1454°C. This means that the parts in the setscrew port were at least 778°C hotter during this run than during the previous high pressure run. Also, the temperature at the outer diameter of the heater base was at least 372°C hotter during this run than during the high pressure run. The higher temperatures experienced for this low pressure run are attributed partly to the lower cooling capability of the low density gas and partly to abnormal heating in the clamp area resulting from the partial fractures in the tongues. The partial fractures at the base of the tongues, if they existed during the heating period, most likely resulted in a local increase in electrical resistance and, hence, led to increased local resistive heating which, in turn, led to higher than normal temperatures in the region. Because of the possibility of abnormally high heating resulting from the fractured tongues, the lower bound of 1454°C for the peak operating temperature does not necessarily apply to a normal low pressure run for which the peak operating temperature may be significantly lower. The melted graphite, noted earlier on the fracture surfaces of the tongues, indicated that the temperature of the graphite in these locations reached or exceeded 3704°C <6700°F> which is the lowest melting point of carbon at a pressure of 2000 psi or higher (below 2000 psi carbon sublimates). It is believed that the temperature at the measurement points did not reach or exceed 2200°C <4000°F> because no boron carbide was observed where the boron nitride housings were in contact with the graphite material.

VII. CONCLUSION

Fusible temperature indicators, in the form of commercial fusible paints as well as fusible metallic ring-shaped samples, were used to determine upper and lower bounds for the peak operating temperatures of a clamped connection between a carbon-carbon heating element and its graphite support base in the Tunnel 9 gas heater vessel.

It was shown that the fusible indicators are little affected by ambient pressure, even up to 22,000 psi, however chemical reactions and eutectic melting effects must be known in order to obtain accurate results. Early testing experience showed that the commercial paints frequently did not

perform reliably when used directly on the hot graphite parts and in the carbon laden atmosphere. Hot pressed boron-nitride and type 304 stainless steel were used as crucible materials to hold the fusible samples and the BN was also used to isolate the samples from electrified graphite parts and from chemically reactive carbon in the Tunnel 9 heater environment. The BN material performed adequately in this regard. The use of aluminum as a melting sample proved unsatisfactory because the small samples were able to retain their original ring shape well beyond the melting point of the aluminum alloy. This phenomenon is partly attributed to the ability of the natural aluminum oxide coating on the sample to maintain the structural shape of the small sample even when the core material is molten. In addition, a non-pure aluminum alloy (type A96061) was used which contained small amounts of other elements one of which may have reacted with the nitrogen gas in the heater vessel and formed a heat resistant film on the sample. The copper and stainless steel samples worked satisfactorily when used with the protective BN crucible. The "Omegalag" 677°C and 954°C melting paints appeared to work satisfactorily when they were held in a stainless steel crucible inside a protective BN shell.

By using the fusible indicators in the Tunnel 9 heating system it was demonstrated that for an off-design operating condition the temperature at the connection between the carbon-carbon heating element and its graphite support base exceeded 1454°C <2650°F>. Also, it was demonstrated that the temperature of the setscrew ports in the graphite support base exceeded 1454°C. Therefore, the tension bolts and washers for the new carbon-carbon heating element clamp should not be made of nickel based superalloys such as INCONEL X-750 because, though the superalloys may work well under the normal operating conditions of the heating system, for off-design conditions the temperature at the clamp connection can reach or exceed the melting range of this and other common nickel based superalloys.

Since the completion of this work, it has been learned that specially developed fusible temperature indicators were used to obtain peak temperature profiles through the thickness of a graphite nozzle liner for a rocket motor¹. The temperature indicators were compositions of metal-carbon and metal-boride-carbon eutectic and peritectic powders usable directly on graphite up to a temperature of 3445°C <6233°F>. The sensors achieve high accuracy by utilizing the fact that the eutectic mixtures with carbon melt at an already depressed temperature, and therefore no further depression is possible. Sensors based on this system show promise as a viable means to measure the peak temperature of the graphite heater parts that operate in the temperature range from 1732°C up to 3445°C.

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TABLE 1 - VARIATION OF MELTING POINT WITH PRESSURE

Material	ΔT_m (°C/ksi)	Comments
Iron	+0.24	avg 0 to 40 kb*
Copper	+0.32	avg 0 to 80 kb
Aluminum	+0.36	avg 0 to 46 kb
Sodium	+0.34	avg 0 to 20 kb
Magnesium	+0.48	avg 0 to 45 kb
Lead	+0.44	avg 0 to 45 kb
Helium	+1.03	avg 0 to 1 kb
Ice (H ₂ O)	-0.70	avg 0 to 2 kb

* 1 kb = 1 kbar = 14,504 psi

TABLE 2 - MELTING AND BOILING POINTS OF SELECTED METALS

Material	Melting Point (°C)	Boiling Point (°C)
Iron	1535	2750
Stainless Steel	1426 (min)	--
Copper	1082	2566
Aluminum	660	2466

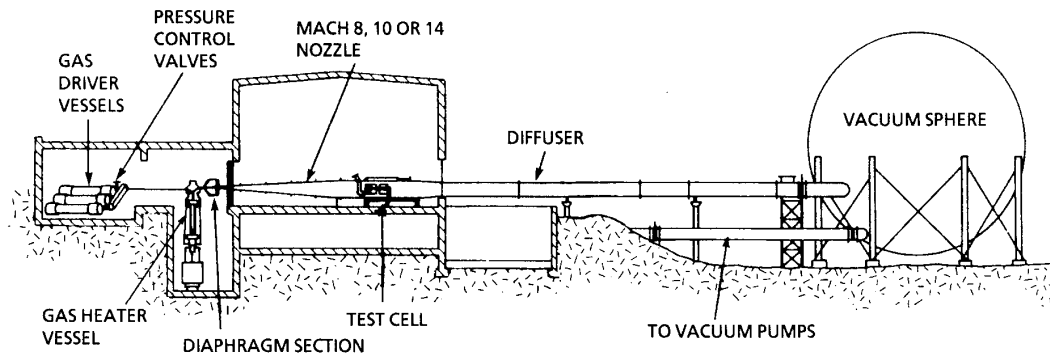


Figure 1. NAVSWC Hypervelocity Wind Tunnel No.9.

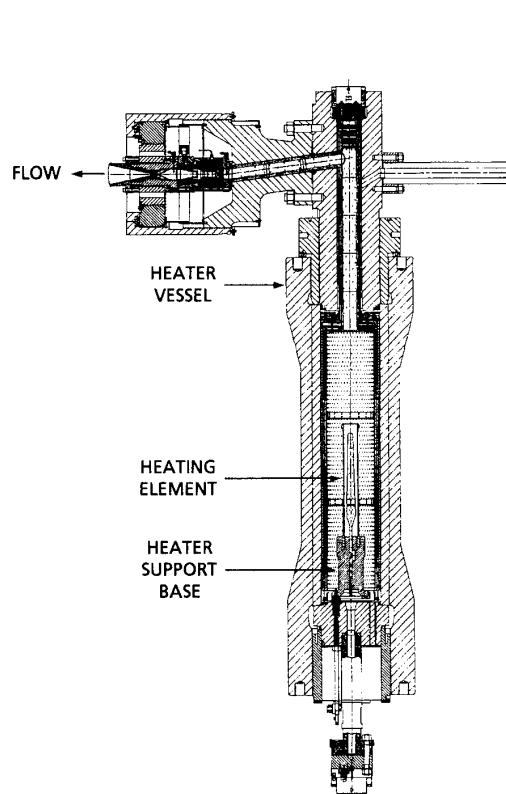


Figure 2. Vertical Heater Vessel and Diaphragm Section.

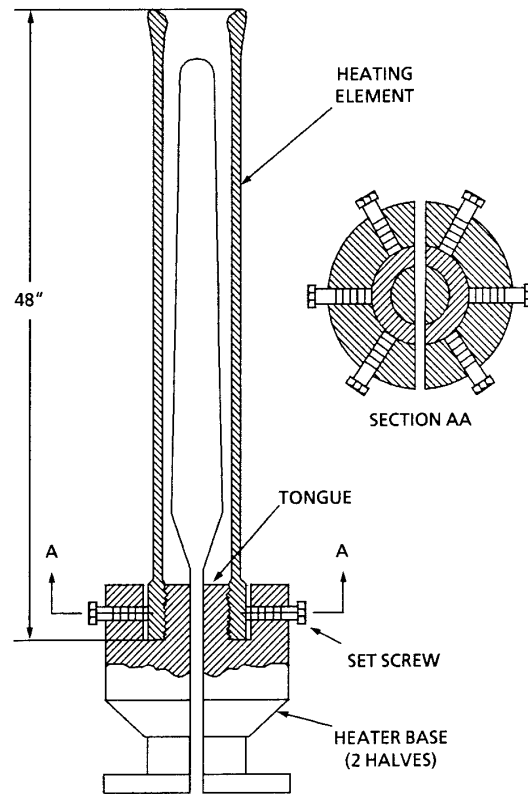


Figure 3. Clamping arrangement for Heater Element and Base.

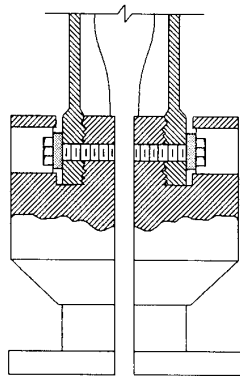


Figure 4. New heater clamp concept.

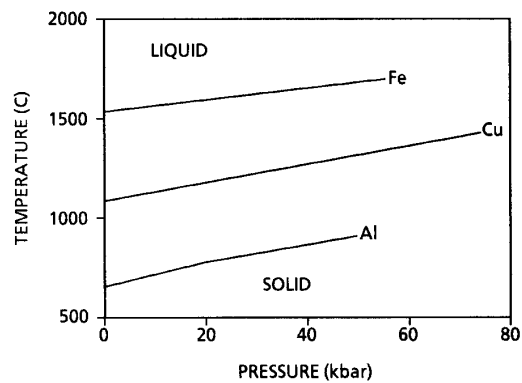


Figure 5. Melting curves for iron, copper, and aluminum.

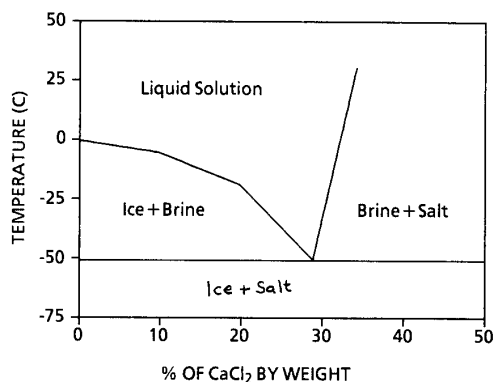


Figure 6. Phase diagram for H_2O and $CaCl_2$ system.

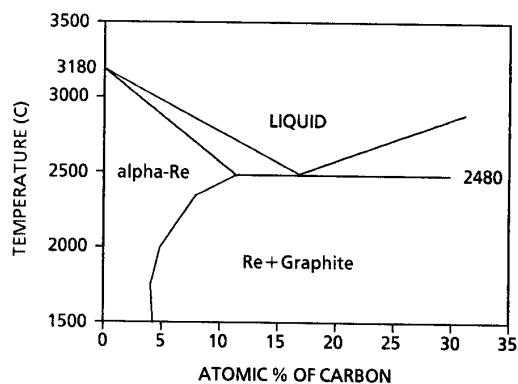


Figure 7. Phase diagram for rhenium and carbon system.

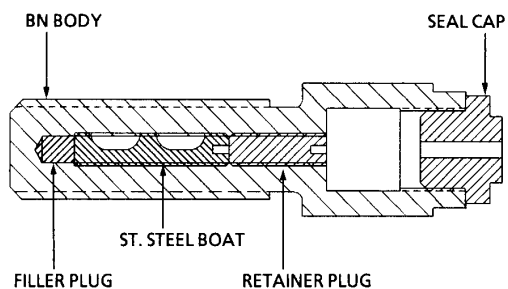


Figure 8. Temperature indicating setscrew.

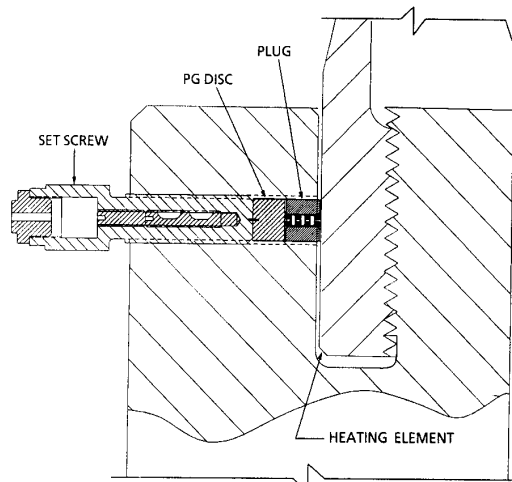


Figure 9. Installation in graphite heater base of temperature indicating setscrew and plug.

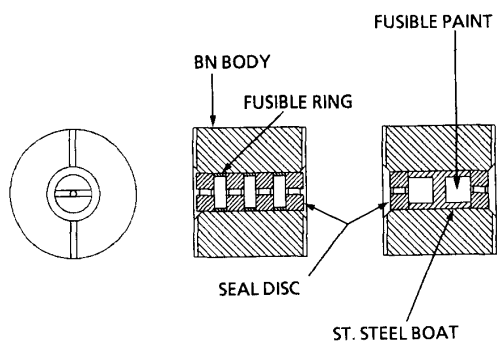


Figure 10. Temperature indicating plugs.

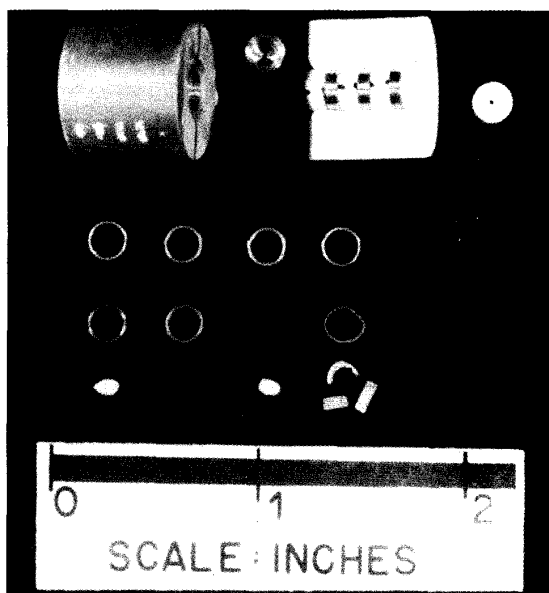


Figure 12.

Temperature plugs, at top, after one tunnel run. Plug at top right is sectioned to show assembly details. Metal ring fusible samples at bottom. First row shows virgin samples. Second row shows steel, copper and aluminum (l. to r.) after a high pressure run. Third row shows resolidified steel and brass pellets and pieces of aluminum ring. The pellets formed during a low pressure tunnel run.

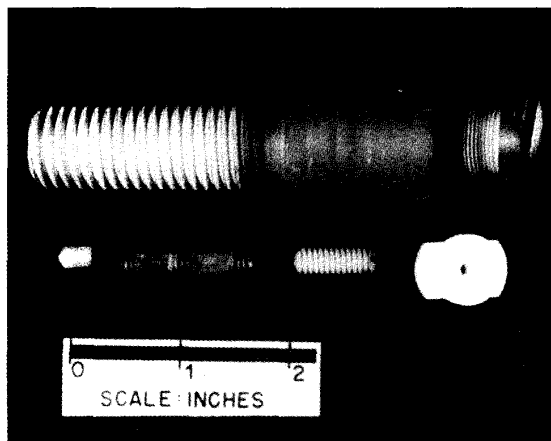


Figure 11.

Temperature indicating setscrew shown disassembled after being subjected to a single high pressure wind tunnel run. White cap at lower right shows virgin BN for comparison.

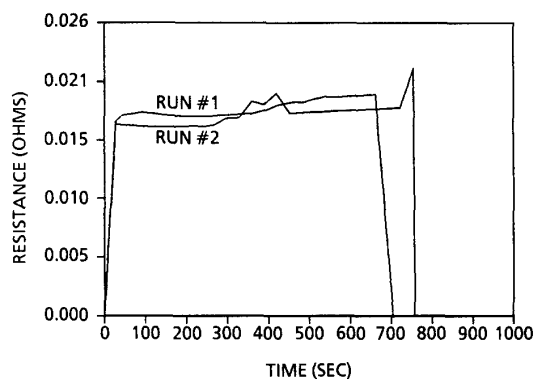


Figure 13.

Heater resistance versus time for the gas heating process for a high pressure (run #1) and low pressure (run #2) tunnel run.