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DRAGON

Dragon Project Report

EARLY WORK ON THERMOCOUPLES TO MEASURE GRAPHITE TEMPERATURE OF 800°C AND HIGHER

by

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ABSTRACT:

A chromel vs. alumel thermocouple sheathed with a titanium stabilized 18/8 austenitic stainless steel has been selected to measure graphite temperatures up to 1100°C in the 7-element rig at C. A. Parsons Ltd., Newcastle.

This report describes the basis of selection, test results and final calibration. Some miscellaneous work on high temperature thermocouples is also included.

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1. INTRODUCTION

This report describes the thermocouple programme carried out in the Engineering Physics Group of the Dragon Project. Most of the work was directed towards the selection of a metal sheathed thermocouple for the measurement of graphite temperatures up to 1100°C for a period of 1000 hours or more.

This work has also served as the basis for a more extended programme on the so called low temperature thermocouples for the Dragon Reactor. This programme is not yet completed and will be described elsewhere. A chapter is devoted to the calibration of these couples for the purpose of control. Another chapter describes miscellaneous work, carried out in the 1500°C - 2500°C temperature region.

2. MEASUREMENT OF GRAPHITE TEMPERATURES UP TO 1100°C

2.0 Description of the Test Apparatus

Three resistance heated horizontal tube furnaces were constructed using nichrome wire and having a maximum operating temperature of 1250°C. For simplicity, the furnaces were controlled directly by means of variacs and the temperatures being measured using a recorder only. This resulted in a slight temperature cycling of plus and minus 20°C around the nominal temperature as a result of a variation of the main voltage. The average period of such a cycle was 6 - 12 hours.

The furnace tube was made of impervious recrystallised alumina. This material does not react with graphite up to 1400°C and was therefore acceptable for the present purpose.

An inert gas atmosphere of very high purity was obtained from a 99.995% pure argon bottle via a purification unit comprising a deoxidant manganous oxide column at 150°C in series with silicagel and molecular sieve driers. The metal specimen or thermocouples were always tightly packed in blind holes, drilled in a cylindrical block of Morgan's graphite EYX 60 (unimpregnated; diameter 28 mm; length 50 mm.).

Both ends of the furnace tube were closed by means of specially made glass caps, containing gas inlet and outlet passages and thermocouple seals, and attached using screw-ring type vacuum couplings.

A gas wash bottle before and after the furnace tube served as a check on the leak rate in the furnace and end caps. This system proved quite satisfactory. During a 1000 hour test no more than 250 litres of argon were consumed with a continuous visible gas flow. There was no detectable weight change of the graphite-block before and after the test which is a check of the high purity of the atmosphere inside the furnace.

2.1 Selection of the Thermocouple Wires

In practice the choice was restricted here to the following two combinations: platinum vs. platinum - 13% rhodium or chromel vs. alumel. Tests showed that a bare thermocouple of pt/pt - 13% rh wires, enclosed by twin hole alumina beads and with the hot junction in direct contact with the graphite is fully reliable for measuring graphite temperatures up to 1200°C for at least 1000 hours. Above 1200°C the platinum forms an eutectic with minor traces of silicon present in either the insulant or the graphite, with a resultant very steep fall in the melting point from 1750°C to 800°C (1). This arrangement was therefore adopted as a standard device up to 1200°C for measurement of the graphite block temperature.

The possibility of using bare platinum vs. platinum - 13% rhodium thermocouple wires to measure the graphite surface temperatures of the single and seven element rigs at Newcastle was rejected on the grounds that settlement of graphite dust between the alumina beads may cause shorting of the thermocouple wires.

Platinum and platinum-rhodium wires serving as the thermo-elements in a metal sheathed type thermocouple appeared to be so embrittled as a result of the alternatively swaging and annealing treatments during manufacture, that such a thermocouple cannot be regarded as a feasible and reliable temperature device.

Chromel vs. alumel therefore remained the only alternative. A bare wire thermocouple of this type cannot be used to measure graphite temperatures because of carburization of the two wires above 900°C (2) with a resultant drift in calibration. However, when enclosed in a protective metal-sheath it proved reliable up to 1100°C for periods exceeding 1000 hours.

2.2 Selection of the Insulant

Three insulants can be considered, beryllia, alumina and magnesia. (3).

The best insulation properties are offered by beryllia but the extreme toxicity of beryllium compounds at high temperatures makes it undesirable for general use. Above 1500°C, however, its electrical resistivity is better than any other known insulant and is therefore often used in special cases as an insulator for very high temperature thermocouples.

Comparatively the least electrically resistant of these oxides, alumina, is widely used in the form of single or twin holed beads for bare thermocouples because of their good mechanical properties. Alumina cannot however be used in a metal sheathed type chromel/alumel thermocouple, because the hardness of the alumina powder results in breakage of the comparatively soft chromel/alumel wires during the drawing and swaging down stages in the manufacturing procedure.

The remaining choice, magnesia, has proved to be the most suitable in the manufacture of metal sheathed chromel/alumel thermocouples and is second only to beryllia in insulation properties. It can be obtained in high purity powder form and even in metal sheathed thermocouple cable with an outer diameter of 0.5 mm. it gives satisfactory service up to 1000°C. The cable has to be sealed off properly, because magnesia is hygroscopic.

The present work has been restricted to thermocouple cables with an outside diameter of 1.5 mm, where the insulation between the wires and the sheath is in the order of mega-ohms. The insulation between the two wires themselves, which is less critical because of the cross sectional geometry of the cable, has never failed during any of the furnace tests.

2.3 Selection of the Metal Sheath

It was mainly in the selection of a metal sheath having satisfactory compatibility with graphite at 1100°C for at least 1000 hours, that a series of furnace tests played a decisive role in the determination of the most suitable thermocouple.

Although restricted to the available metal sheaths, owing to the short time scale, the experimental programme covered all available metal or alloy sheaths likely to prove suitable; e.g. nickel; copper-nickel alloys; stainless steels and inconel.

2.3.1 Nickel

The sheath material available was a high purity nickel of small grain size. (Fig. 1.) All tests between 1000°C and 1200°C running for 60 hours and 300 hours, showed a very marked grain growth (Fig. 2). The solubility of graphite in nickel is rather high at these temperatures, 0.25 weight % at 1000°C (1), and strongly temperature dependent. This, combined with the above mentioned slight temperature cycling explains the marked carbon precipitation at the grain boundaries. The combination of grain growth and carbon precipitation embrittled the sheath considerably even during the preliminary 60 hour tests.

An attempt to stop carbon diffusion into the nickel by coating the nickel sheath with a layer of alumina, approximately 0.1 mm. thickness, had no effect because of the porosity of such a coating.

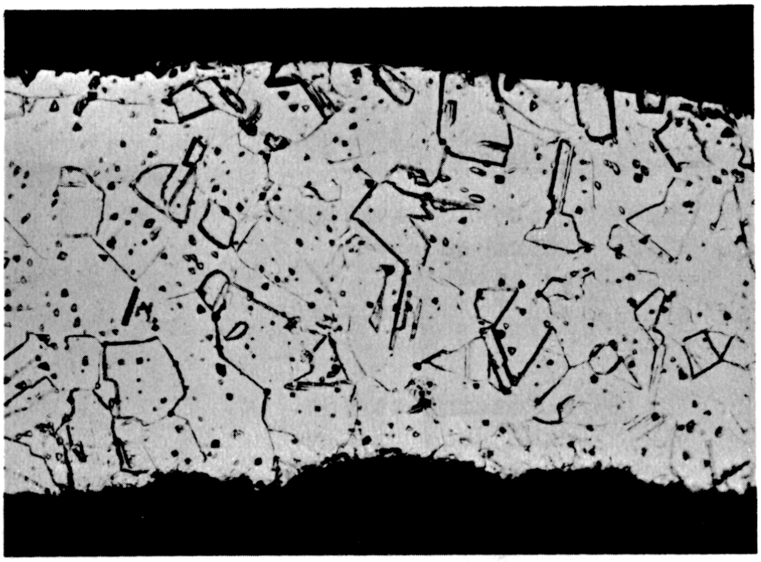

Figure 1. High Purity Nickel Sheath, Small Grain Size, as received (300 x)

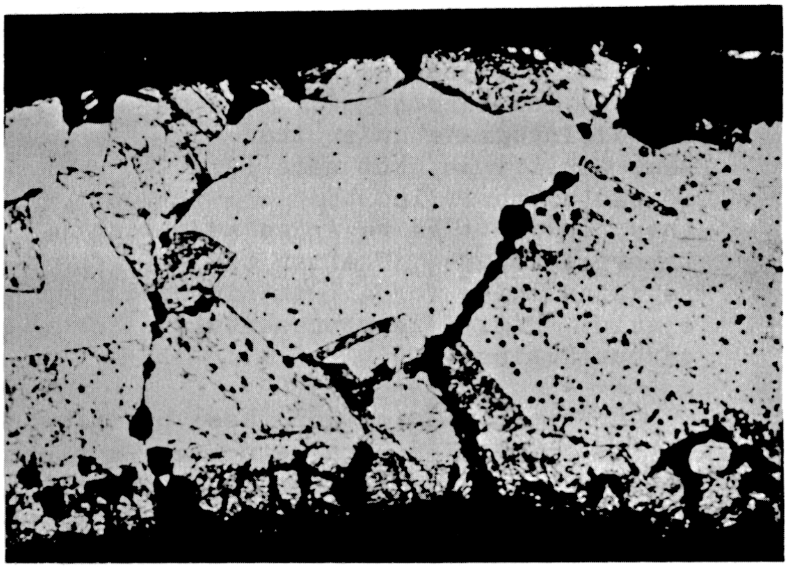

Figure 2. High Purity Nickel Heated in contact with Graphite for 300 hours at 1100°C. (300 x)

2.3.2 Copper - Nickel Alloys

Copper and nickel alloy well over the whole range from 100% copper to 100% nickel. 100% copper would be ideal since it has extremely good compatibility with graphite, it does not form carbides and creates an ideal diffusion barrier as the solubility of carbon in copper is only 0.001 weight %, at 1000°C (1). Unfortunately, however, its melting point, 1083°C, precludes its use for temperatures exceeding 1000°C. The only available copper-nickel alloy obtainable in the form of a thermocouple sheath was 70% copper, 30% nickel. The melting point of this particular alloy is above 1150°C. Under test it failed due to local melting.

Tests at General Dynamics (4) have shown that a copper-nickel alloy, known as "monel", consisting of 70% nickel and 30% copper with a melting point of approximately 1300°C, gives excellent compatibility with graphite at 1000°C. Unfortunately this material is not yet commercially available in the form of thermocouple sheaths.

2.3.3 Stainless Steels

Three types were available: 25/20 stainless steel; 18/8 stainless steel and stabilized 18/8 stainless steel.

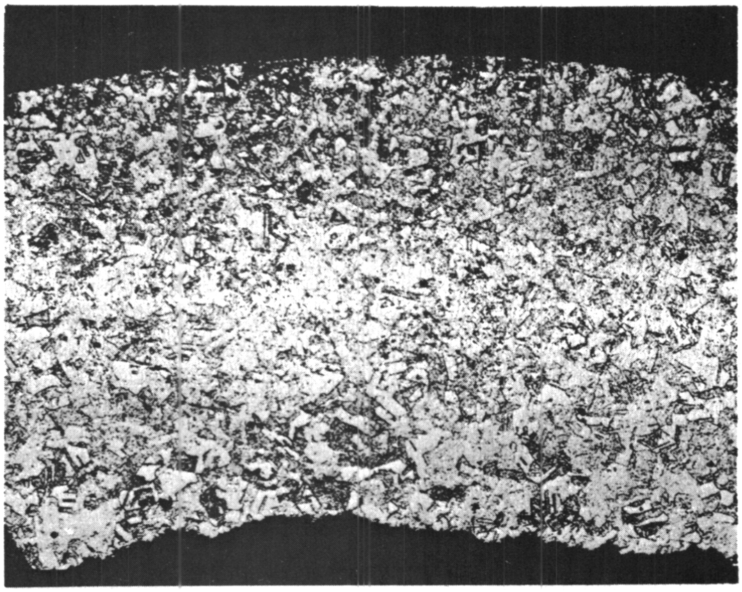

Figure 3. 18/8 Type Austenitic Stainless Steel (as received) (300 x)

The 25/20 stainless (St. St. 310) formed such massive carbides due to the high chromium content, 23.5%; that the material disintegrated completely after 300 hours at 1100°C in contact with the graphite. The investigated 18/8 stainless steels (St. St. 347, St. St. 304 and St. St. 304 low carbon) did not disintegrate under the same conditions, but were so badly embrittled that they could not be regarded as reliable. Fig. 3 and Fig. 4 show the material before and after the 300 hour test at 1100°C. Fig. 3 is representative for all the investigated austenitic 18/8 type stainless steels; Fig. 4 is representative for these stainless steels after carburisation.

After carburisation these pictures always show two phases: one is the original carbon saturated, austenite; the second is the hard phase of the chromium carbides (5). The carbide phase occupies approximately 50% of the structure which explains its extreme brittleness. Of the 18/8 type austenitic stainless steels with a stabilizer (titanium niobium, or columbium) only the stainless steel with a titanium stablizer (St. St. 321) could be obtained at the time. However, no difference between the various stabilised stainless steels has been reported. The mechanism by which the stabiliser acts is assumed as follows: carbon, originating from either solid graphite or carbon monoxide, no longer attacks the chromium with a resultant chromium carbide precipitation and breaking up of the original alloy composition; but is now caught by the stabilizer, forming also a carbide but leaving the original alloy composition intact (6). A marked improvement in compatibility was obtained with this stabilized stainless steel (Fig.5); after 300 hours at 1100°C the sheath was still reasonably ductile. However, after extending the test period to 1000 hours, a structure very much the same as that of the other stainless steels was obtained (Fig. 6) and although the sheath kept its integrity a severe embrittlement had occurred. It would seem that after the complete carburisation of the stabilizer, carburisation of the stainless steel follows the same mechanism as with the unstabilised 18/8 stainless steels. The main conclusion of these tests is that a stabilizer serves only to delay the carburisation of the steel in the presence of bulk graphite. The titanium stabilised 18/8 austenitic stainless steel sheathed thermocouple showed no variation in signal during the 1000 hour test; but it is believed that it was approaching its limit of endurance.

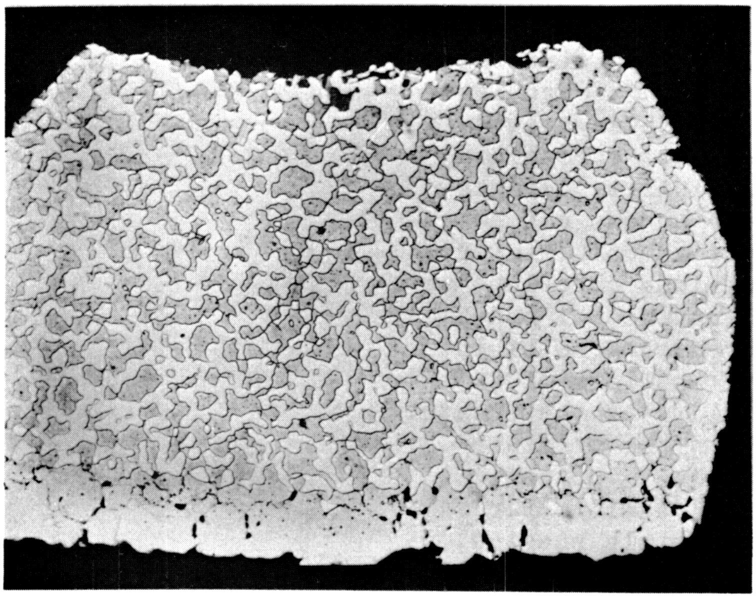

Figure 4. 18/8 types Austenitic Stainless Steel after 300 hours at 1100 C in contact with Graphite. (300 x)

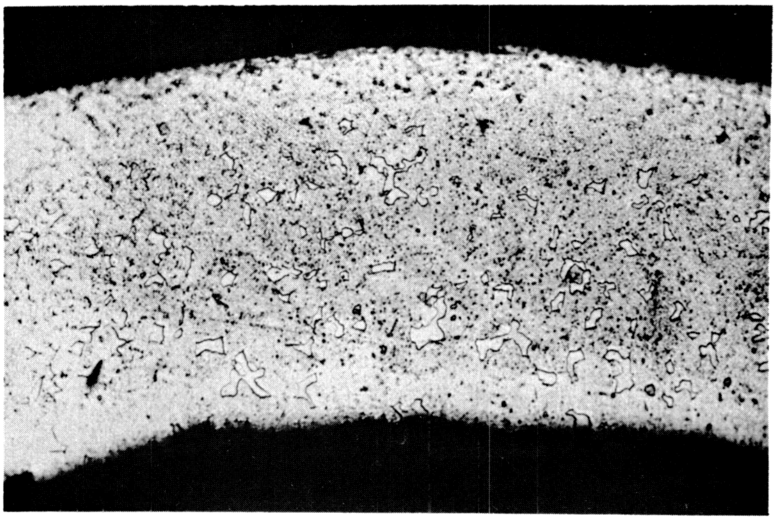

Figure 5. 18/8 type Austenitic Stainless Steel Titanium Stabilized after 300 hours at 1100°C in contact with Graphite. (300 x)

An 18/8 type stainless steel with a chromised surface layer showed no advantage regarding compatibility because of the porous nature of the chromium-layer. Impervious chromium-plating was not considered because of the resultant loss of ductility of the sheath. Other types of stainless steels do not appear feasible for thermocouple sheaths, mainly because of brittleness.

2.3.4 Inconel

The fourth group of investigated sheath materials consisted of three inconel sheaths, all having approximately the following composition: 15% chromium; 8% iron and 76% nickel.

Figure 7, Figure 8 and Figure 9 present the inconel sheath before testing and after a 300 hour and a 1000 hour test at 1100°C. The two phases in Figure 8 are presumably the original, carbon saturated, alloy and again a hard chromium carbide phase. The rate of carbide formation and hence embrittlement appears to be about the same to that of the stabilised stainless steels at these temperatures. The reported better compatibility for inconels compared to stainless steels versus graphite at approximately 900°C

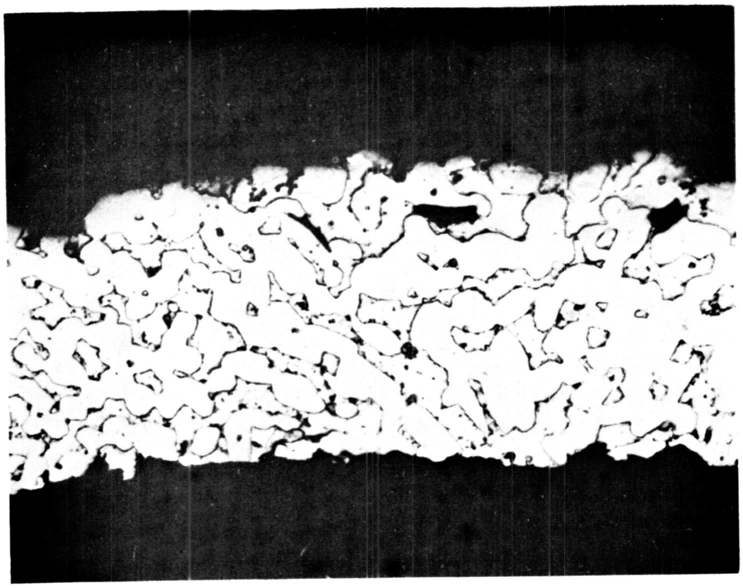

Figure 6. Idem as on Figure 5, but after 1000 hours. (300 x)

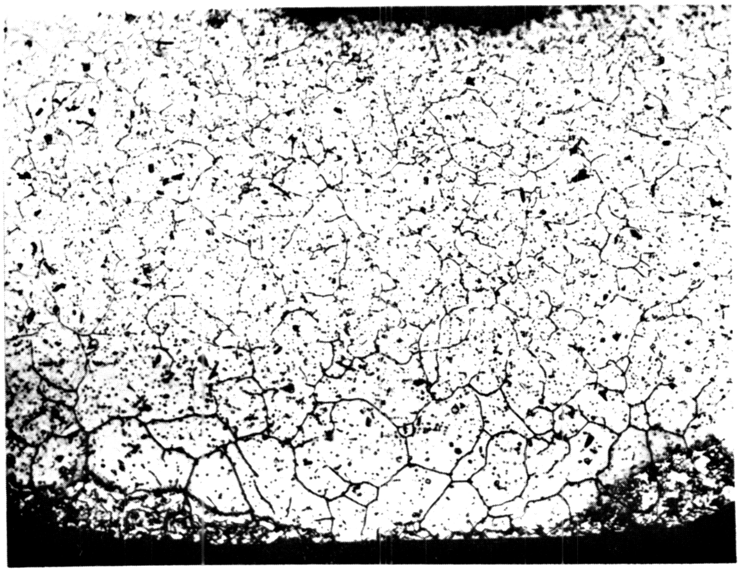

Figure 7. Inconel (as received) (300 x)

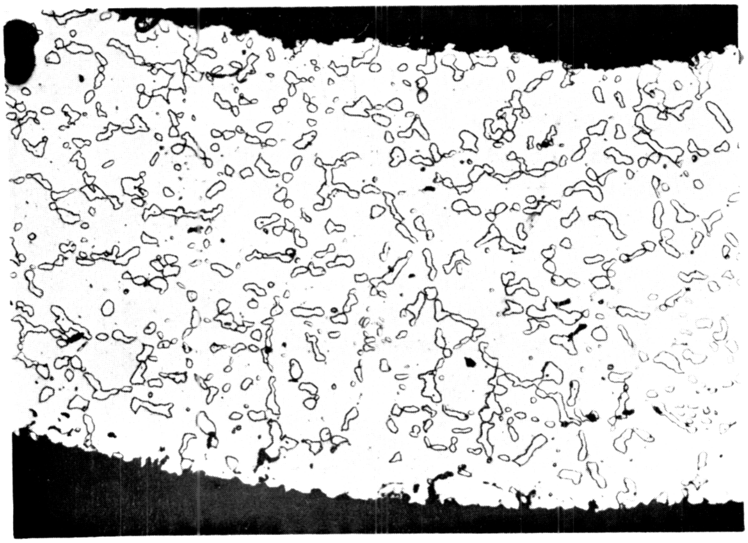

Figure 8. Inconel after 300 hours at 1100°C in contact with Graphite. (300 x)

(7, 8) were not confirmed by our results at 1100°C insofar as stabilized stainless steels are concerned.

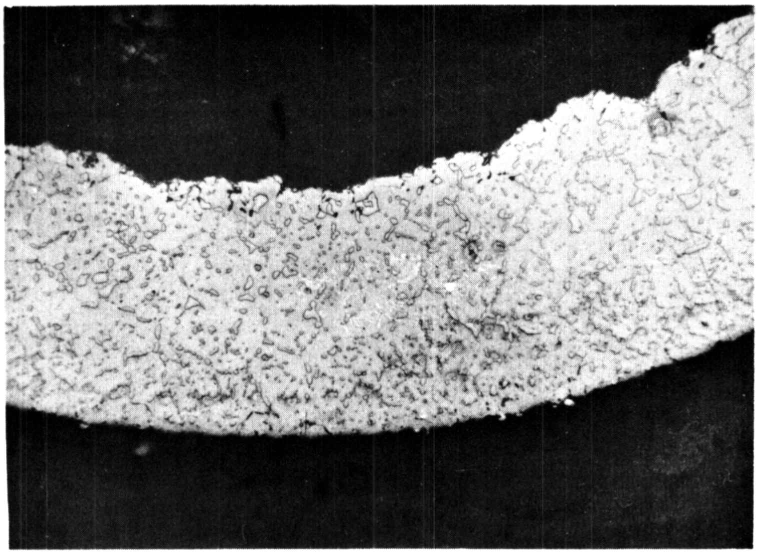

Figure 9. Inconel after 1000 hours at 1100°C in contact with Graphite. (300 x)

Finally attention is drawn to the following qualitative observations made during the tests.

Firstly in cases where the inert furnace atmosphere was contaminated with oxidising impurities, coming off from the walls or as a result of imperfect outgassing of the graphite, it was noted that at temperatures of about 1000°C, inconel suffers less from surface oxidation than stainless steel. This surface oxidation phenomena is difficult to understand, since all the oxidising impurities should react with the solid graphite to form carbon monoxide.

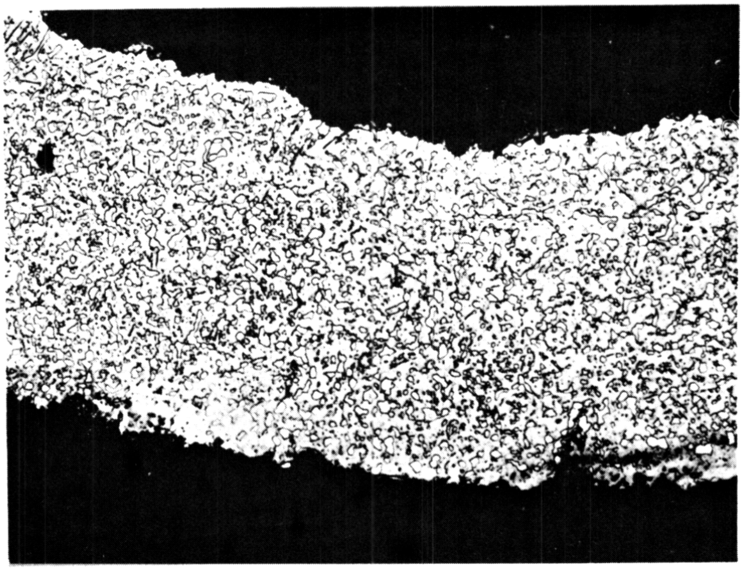

Figure 10. 18/8 type Austenitic Stainless Steel titanium stabilized after 1000 hrs., suffering from Carbon Monoxide attack causing carburization at temp. 500°-800°C. (300 x)

The second point is, that serious carburisation and embrittlement of the sheath was also occurring downstream, where the sheath was exposed only to the furnace atmosphere at temperatures well below 1000°C (Fig. 10). The carbon monoxide concentration in this region could not have been higher than a few hundred v.p.m., because there was no measureable weight loss of the graphite. The carbon atoms in the carbon monoxide do, however, carburise a metal much easier than carbon atoms coming from the solid graphite, due to the steel catalyzed back reaction $2CO \rightarrow CO_2 + C$. A slight carbon dust deposit on the sheaths in the temperature region 400°-800°C, was also normally observed after the tests, presumably caused by the same reaction.

The third point is illustrated by Fig. 11. In this picture a peculiar attack of the inside of the sheath is apparent which only sees the magnesia insulant. This attack has been observed on various thermocouples and appeared to have no relation to our tests. It is almost certainly a sulphur attack. The sulphur presumably originates from residual traces of drawing compounds used during the manufacture of the sheathing tube. During the first stage of annealing, which takes place at approximately 900°C, these residues will attack the inside of the sheath. Only in one sample did this attack penetrate through the entire sheath-thickness (0.15 mm.). In the other cases it was only casually observed and the depth of penetration was only a few thousandths of an inch.

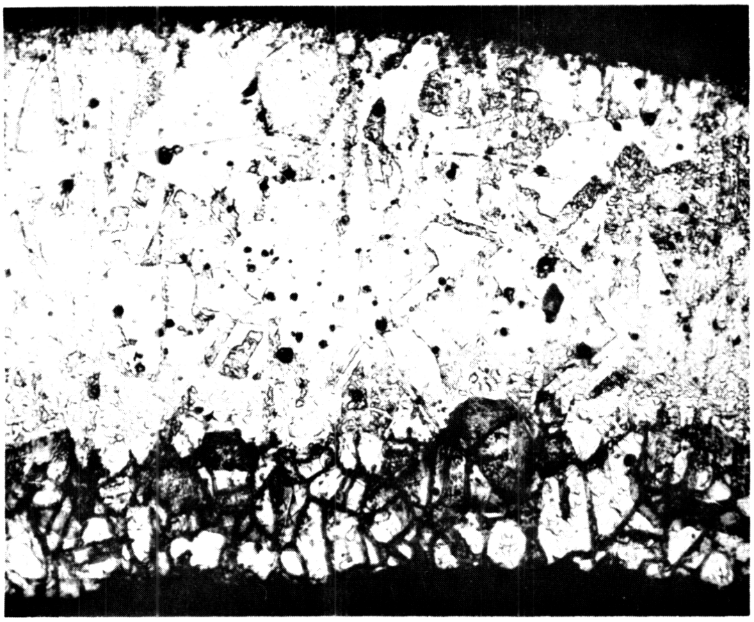

Figure 11. Pure Nickel Sheath as received, with Sulphur attack at the inside of the Sheath. (300 x)

2.4 Discussion of the Results

It was concluded that no completely reliable thermocouple sheath was available for operation in graphite at 1100°C for much more than 1000 hours. Fortunately the bulk of the graphite in the Dragon Reactor is only 800°C and for these conditions the selection of a compatible metal sheath is very likely to result out of the extended "low temperature" thermocouple programme.

Apart from the risk of disintegration of the sheath with a subsequent rapid destruction of the whole thermocouple, there is another cause of failure of a slightly different nature. If the sheath is not a good diffusion barrier, because too much carbon can go into solution with the sheath material and hence diffuse through it, then there will be a slow but constant contamination of the magnesia insulant at the inside of the sheath. After the 1000 hour tests all thermocouples showed an enormous fall in resistance between the wires and the sheath, from several megaohms to several hundreds of ohms and in some cases even to several tens of ohms. A slight colour change of the magnesia could be observed in some cases to illustrate this diffusion effect. Although this mechanism did not produce shorting between the thermocouple wires during 1000 hours, it is estimated that this would have occurred in due course. This underlines the point, that it is not only the compatibility that must be considered in selecting a good sheath, but also the amount of carbon that can be in solution in the sheath material, which determines the rate at which the carbon will diffuse through the sheath.

Tests carried out to determine the best method of attachment of the thermocouple onto the surface of the graphite fuel rods in the single and seven element rigs will be described in a later report. The same applies for another series of tests, in which large temperature gradients were created along the thermocouple leads, with the hot sensing end at a low temperature and parts of the sheath at very high temperatures. These tests were carried out to check the reliability of the temperature-signal. Although these signals could be slightly affected in this way, the resultant deviation was not significant and full scale operational results must be awaited before any comment can be given on this point.

3. CALIBRATION OF THERMOCOUPLES

3.0 Description of Test Apparatus

The calibration furnace is a vertical tube furnace, which can be heated up to 1100°C. Various materials can be inserted and heated up to their melting point. The calibration of the couples is done by inserting them into a thin walled blind tube, immersed in the melt.

As reference temperatures the freezing points were taken of tin (231.8°C), zinc (419.7°C) and copper (1083°C). The freezing points are more reliable than the melting points because in the liquid metal there is a better thermal contact between the various parts involved and therefore a greater guarantee, that no temperature gradient in the melt was existing at the time of recording the reference temperature (9).

The resultant potentials from the couples were recorded on a high precision potentiometer, and then compared with the given British Standard Specifications for these couples.

3.1 Discussion of the Measurements

Contamination of the zinc and the copper point invalidated these results.

The steel tube, containing the couples reacted with the zinc - and will be replaced by a blind ended alumina tube. The copper surface oxidised and showed a shift in the melting point. This will be eliminated by containing the copper in a graphite crucible and protecting the surface with graphite powder.

Of various stainless steel sheathed chromel/alumel thermocouples; the calibrations tested to date appear to be within the limits prescribed by the British Standards Institution. (12).

4. MISCELLANEOUS

4.1 Description of a 2500°C Furnace

Considerable effort was put into the design, construction and commissioning of a small furnace which can give temperatures up to 2500°C. The heat source consists of a self supported double-helix tungsten spiral. Inside the small spiral, bore 8 mm., is a cylinder of graphite to equalise the temperature. In the centre of the graphite is a hole where a thermocouple specimen can be inserted. A series of radiation shields provide the thermal insulation; as long as the temperature test does not exceed half a day. The whole assembly is installed in a glass belljar, standing on a brass bed-plate through which electrical power, gas connections etc., enter through suitable seals. Temperature measurement is by an optical pyrometer, focussed on the graphite block in the centre of the tungsten coil through holes in the radiation shields.

Due to the complicated geometry of the installation it was found impossible to purge the system thoroughly with inert gas to avoid oxidation of the furnace components. Tests were therefore carried out at a high vacuum, which proved to be a satisfactory alternative. Distortion of the various parts in the hot zone, resulting in short circuits, proved a further source of difficulties which was finally overcome by detailed redesign on the basis of experience and a careful stress-relieving of the hottest components.

4.2 Test of a Graphite vs. Tungsten Thermocouple

The only test so far executed in this furnace has been several calibration runs of a tungsten versus graphite thermocouple. (10). Up to 1650°C each individual couple showed a fairly consistent calibration curve; no hysteresis effect in the heating up and cooling down curves was observed. However, the extreme embrittlement of the tungsten above 1000°C made the whole couple very fragile after high temperature testing.

4.3 Description of a 1500°C Furnace

This furnace is a horizontal tube furnace, made by Gallenkamp Ltd. The impervious recrystallised alumina furnace tube is heated by four molybdenum-disilicide rods, surrounding and parallel to the tube. The temperature of the outside of the tube is recorded by a platinum-rhodium vs. platinum reference couple. A recorder controller maintains the temperature automatically at a preset value by means of an on-off control of a variable transformer. The furnace is designed for maximum temperature operation of 1550°C and has actually performed very well at 1500°C for several hundreds of hours.

4.4 Test of a Series of Thermo-elements at 1500°C

Six thermocouples consisting out of all possible combinations of tungsten, tantalum, tungsten-rhenium and molybdenum, were tested for 300 hours at 1500°C (11). The same precautions were taken for this test as for the 1100°C tests. Molybdenum foil was wrapped around the graphite block in order to stop diffusion from the carbon into the alumina furnace tube. The result from that test was erroneous. Inspection of the furnace at the end of the test showed that all the materials suffered badly from both carburisation and oxidation, although the total impurity contact would not have exceeded 1000 v.p.m. of carbon monoxide, estimated from observed carbon mass transfer.

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When this test was repeated by Mr. G. W. Horsley in an induction heater at a very high vacuum (10^{-5} mm. Hg.) the results were entirely different. The conclusion is, that at temperatures of 1500°C and higher the compatibility behaviour of these metals with the graphite are largely governed by the carbon monoxide concentration, which is a function of the initial impurity of the inert gas atmosphere and further outgassing of the graphite.

Considerable effort was, therefore, devoted to obtaining a controllable and constant atmosphere inside a furnace by means of a closed gas circuit. The new test set-up is now in the commissioning stage and the description of it falls outside the scope of this report.

5. CONCLUSIONS

The work described in this report will serve as a basis for the ultimate selection of a thermocouple to measure graphite temperatures up to 800°C in the Dragon Reactor. This 800°C temperature limitation is appreciably lower than the 1100°C limitation for the single and seven element rigs. This advantage is however off-set by the fact that the couples in the Dragon Reactor up to 800°C have to serve a life-time of at least two years: whereas the couples in the rigs have only to serve two months.

Valuable experience with regard to the full scale installation and operation of these couples will be obtained from these rigs.

With regard to the high temperature couples up to 1500°C the programme is still in its early stages and no consistent results can be reported yet.

6. ACKNOWLEDGMENTS

Special acknowledgment is due to Mr. E. W. V. Acton, now at C.E.G.B. Heat Transfer Laboratories, Berkeley, Gloucestershire, under whose guidance this work was started.

Also to Mr. E. H. Davies, for carrying out most of the experimental work, in which he was assisted by Mr. Underhill.

Acknowledgments are also due to Mr. Day of Fulmer Research Institute, who did part of the metallurgical examinations and to members of the Dragon Materials Division for their support and advice at various stages of this work.

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