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THERMAL CAPSULE CALIBRATIONS

(Title Unclassified)

A. Filippi

June 30, 1964

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74

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TABLE OF CONTENTS

		<u>Page</u>
I	Abstract	1
II	Introduction	1
III	Equipment and Calibration Procedure	2
IV	Results and Discussion	4
V	Conclusions	12
VI	Recommendations	13
VII	References	14

LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	Calibration Results for WANL Wire Code Number 1	15
2	Calibration Results for WANL Wire Code Number 2	16
3	Calibration Results for WANL Wire Code Number 3	17
4	Calibration Results for WANL Wire Code Number 4	18
5	Calibration Results for WANL Wire Code Number 5	19
6	Calibration Results for WANL Wire Code Number 6	20
7	Calibration Results for WANL Wire Code Number 7	21
8	Calibration Results for WANL Wire Code Number 8	22
9	Calibration Results for WANL Wire Code Number 9	23
10	Calibration Results for WANL Wire Code Number 10	24
11	Calibration Results for WANL Wire Code Number 11	25
12	Calibration Results for WANL Wire Code Number 12	26
13	Calibration Results for WANL Wire Code Number 13	27
14	Calibration Results for WANL Wire Code Number 14	28
15	Calibration Results for WANL Wire Code Number 15	29
16	Calibration Results for WANL Wire Code Number 16	30
17	Calibration Results for WANL Wire Code Number 17	31

LIST OF TABLES
(Continued)

<u>Table</u>		<u>Page</u>
18	Calibration Results for WANL Wire Code Number 18	32
19	Calibration Results for WANL Wire Code Number 19	33
20	Calibration Results for WANL Wire Code Number 20	34
21	Calibration Results for WANL Wire Code Number 21	35
22	Calibration Results for WANL Wire Code Number 22	36
23	Calibration Results for WANL Wire Code Number 23	37
24	Calibration Results for WANL Wire Code Number 24	38
25	Calibration Results for WANL Wire Code Number 25	39
26	Calibration Results for WANL Wire Code Number 26	40
27	Calibration Results for WANL Wire Code Number 27	41
28	WANL Thermal Capsule Code Numbers, Composition, Predicted Melting Temperature, and Calibration Temperatures	42

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Thermal Capsule and Cassette Construction 1X	43
2	Close-up Photograph of Heating Unit	44
3	Photograph of Calibration Equipment	45
4	Microstructural Cross-Section Through Sample 25AA	46
5	Phase Diagram of the Tantalum - Carbon System	47
6	Radial Carbon and Tantalum Gradient in Sample 25AA	48
7	Cross Sectional Structure Through Tantalum Thermal Capsule Wire Showing Once-Molten Core	49
8	Cross Sectional Structure Through Tantalum Thermal Capsule Wire Showing Completely Carburized Structure	50
9	Microstructure Characteristic of Once-Melted Areas in Codes 19, 21, 22, 24 and 25	51

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
10	Point and Radial Body Microstructures for Various Code 19 Samples (21 w/o Ta - 79 w/o Nb)	52
11	Point and Radial Body Microstructures for Various Code 21 Samples (67 w/o Ta - 33 w/o Nb)	53
12	Point and Radial Body Microstructure on Various Code 22 Samples (78 w/o Ta - 22 w/o Nb)	54
13	Point and Radial Body Microstructures on Various Code 24 Samples (90 w/o Ta - 10 w/o Nb)	55
14	Point and Radial Body Microstructures on Various Code 25 Samples (Pure Tantalum)	56

ABSTRACT

Twenty-seven thermal capsule materials to be used in determining NERVA core temperatures have been calibrated. These cover the range of temperature from 660° to 2800°C, and consist of 11 metal-graphite combinations chosen by WANL and 16 chosen by LASL. (A thermal capsule consists of a 0.040" diameter x 1/4" long pointed metal wire enclosed in a graphite case.) Calibration was performed on 22 combinations by observing the lowest temperature at which the wire point deformed. Difficulty, however, was encountered with this procedure when calibrating five of the high-temperature capsule materials. These wires, four niobium-tantalum alloys and pure tantalum, readily formed surface carbides during calibration which resisted deformation even when internal melting occurred. The detection of melting by observing a change in wire tip geometry was, therefore, inadequate, and instead calibration was accomplished metallographically. All combinations, with one exception, showed melting within $\pm 50^{\circ}\text{C}$ of the predicted temperature.

INTRODUCTION

Early work conducted at LASL encountered difficulty with the use of molybdenum-sheathed thermocouples in contact with graphite. Reaction occurred between the molybdenum and graphite limiting the temperature range of thermocouple reliability. Because of this, the concept of using metal-graphite eutectic temperatures (lowest melting temperature in a given alloy system) as a means for sensing NERVA core temperatures evolved. This procedure involves the placing of a set of metal-graphite combinations at core locations of interest. After hot reactor testing, the temperature at these locations is deduced by determining which combinations had melted and which had not. In principle the idea is similar to the use years ago of pyrometric cones by heat treaters to determine furnace temperatures wherein deformation of the cone signifies melting.

Twenty-seven metal-graphite combinations which show signs of melting at specific temperatures have been chosen for NERVA core temperature-sensing applications. Each combination consists of a 0.040" diameter x 1/4" long pointed metal wire placed inside a graphite case. A picture of a typical metal wire-graphite combination (termed thermal capsule) is shown in Figure 1. A series of thermal capsules is cemented into unfueled NERVA core elements, and a radiograph of these elements is taken to establish a record of the shape of each metal wire. In particular, radiography is performed to fix the configuration of each wire point. After hot reactor testing, a second set of radiographs is taken to determine which wire points are deformed, signifying melting, and which retain their original shape. Through knowledge of the particular wire at each position, and the melting temperature of each wire, the peak temperature at the location can then be deduced.

This report is concerned with temperature calibration of each thermal capsule combination. The specific wire compositions are given in Table 28, and span the temperature range 660° to 2800°C. Each wire has been assigned a WANL code number, also shown in Table 28, and for the remainder of this report reference will be made to these code numbers as well as to specific compositions.

EQUIPMENT AND CALIBRATION PROCEDURE

Calibration of the various combinations was performed on thermal capsules identical to those which will be placed in the NERVA core. The majority of capsules (codes 3 through 27) were calibrated by heating to specific temperatures inside an induction coil followed by visually observing the wire tip for signs of melting. Black body conditions for optical temperature measurements were simulated by placing the capsules inside a graphite cassette (Figure 1).

For a calibration run the capsule-cassette assembly was placed inside a water-cooled copper current concentrator which in turn was positioned in a large quartz tube. A photograph taken through the side of this tube and showing the copper current concentrator surrounding a glowing cassette is shown in Figure 2. The tube was twice

rough-pump evacuated and back filled with helium prior to a run. An induction coil placed around the tube and drawing power from a 10 KVA R. F. induction set provided the necessary energy for heating. The quartz tube, current concentrator, induction coil and cassette configuration were supplied by LASL. Temperature was measured optically through a hole in the cassette lid. The escaping light passed through a glass port and was bent through a prism to allow the operator to easily take temperature readings. A photograph of the calibration equipment is shown in Figure 3. The optical system (the glass port, prism, and optical pyrometer) were calibrated as a unit by the WANL Instrumentations Laboratory using NBS standard lamps. The pyrometer was used solely for thermal capsule evaluation during the period of the program.

A single calibration run consisted of heating a capsule to a specific temperature in less than five minutes followed by holding at temperature for exactly five minutes. Temperatures approximately 50°C below a given calibration temperature were achieved within a few seconds by pre-setting the induction unit power prior to beginning a run. The calibration temperature was then attained by increasing the power in steps, a procedure which took anywhere from 30 seconds to five minutes to accomplish. Time-to-temperature data were not recorded, but were limited to a maximum of five minutes. The thermal capsule lid was removed to enable viewing directly onto the wire tip for temperature measurement. For a given calibration sequence, several capsules per wire code were heated, each to a temperature in the vicinity of the predicted melting point. After cooling to room temperature the wire points were visually inspected* for signs of melting. Calibration runs, varying the temperature, were made until the temperature range between the highest reading wherein melting did not occur and the lowest where melting occurred was narrowed to 20°C or less. The closest five-degree temperature to the average of this range was then reported as the melting temperature of the particular wire. Because induction heating was used for codes 3 through 27 a range rather than a specific

* Visual wire inspection was inadequate for codes 19, 21, 22, 24, and 25.
Metallographic inspection for melting had to be substituted.

temperature resulted during each run. In general, the range did not encompass more than 15°C. When calculating the melting temperature, the highest readings obtained on the runs representing the two which bracketed the melting point were used.

The accuracy of the melting temperature attained as described above is considered to be $\pm 30^{\circ}\text{C}$. Operator errors in optically reading thermal capsule temperatures, and pyrometer errors are estimated to contribute $\pm 20^{\circ}\text{C}$ variance in the calculated melting point. An additional possible 10°C error is superimposed upon this due to the assumption that the melting temperature lies at the midpoint between an unmelted and melted sample which are separated by a maximum of approximately 20°C.

In describing the calibration data, three general tip configuration categories were used: namely, pointed, slightly rounded and rounded. The latter two categories represent deformed configurations due to slight or complete melting while the pointed category represents an unchanged wire tip. The actual wire tip shape after melting depends upon the degree of liquid wetting of the graphite, and varies from concave through convex over the span of wires considered. Therefore, the term rounded is only meant to represent a melted tip rather than defining an actual tip shape.

The experimental procedure used to calibrate WANL codes 1 and 2 was somewhat different from that described above. Because these wires melted at temperatures too low for optical measurement, resistance furnace heating and thermocouple temperature measurement were used. The capsules were inserted in an already heated resistance furnace and protected from oxidation with flowing argon. Temperature measurement was taken from a thermocouple positioned adjacent to the capsule. All thermal capsule calibration runs were witnessed by a WANL Quality Control representative.

RESULTS AND DISCUSSION

Codes 1 through 18, 20, 26, and 27 were easily calibrated by visually examining the wire point on several capsules per code. Data on individual codes are shown in Tables 1 through 18, 20, 26, and 27 along with photographs of some

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of the actual experimental capsules. The degree of wire tip deformation signifying a melted sample was somewhat arbitrary and, in general, the complete loss of a sharp point has been defined as a rounded or melted condition. Sample preparation for photographs was accomplished by filing away the portion of the graphite case adjacent to the point of the wire. The melting temperatures of these, as well as the codes still to be discussed, are compiled in Table 28.

Difficulty was encountered with the calibration of codes 19, 21, 22, 23, 24, and 25. The tungsten wire of code 23 tended to expand out of the graphite case upon melting which led to erroneous temperature readings. In order to prevent this, the thermal capsule cover had to be retained on some samples, and temperature measurements were taken from this cover. Both "cover off" and "cover on" data are presented in Table 23. It can be seen that the more reliable results were obtained with the cover on. The problem which developed during calibration of codes 19, 21, 22, 24, and 25 was of a different nature. These wires developed a series of high-melting-temperature carbide surface layers during calibration. The layers resisted deformation and retained the original wire shape even when melting occurred at the lower-carbon interior sections. Because of this, inconsistent results were obtained with the determination of melting by visually observing wire tips, and a metallographic procedure for detecting once-melted regions had to be worked out.

The easiest wire in this group to analyze to explain what occurred to these materials is code 25, pure tantalum. Figure 4 shows a photomicrograph of the length of a code 25 wire polished to the depth of its diameter and etched to reveal its microstructure. This particular wire had undergone a calibration run at 2747° to 2749°C (sample 25AA, Table 25). During calibration three distinct, separable and concentric layers of differing carbon content occurred from the surface to the center of the wire. The phases, as seen from the phase diagram in Figure 5, consist of the high-melting TaC phase at the surface, Ta_2C immediately under this, followed by a core of tantalum with carbon in solid solution. Figure 4 reveals a white-appearing TaC layer at the outside followed by a basket weave layer of mixed carbides of TaC and Ta_2C ; the

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Ta₂C phase formed due to the decreasing solubility of tantalum in TaC during cooling. The next layer in sequence is white-appearing Ta₂C which is followed by a region of Ta in a Ta₂C matrix formed upon cooling due to the decreasing solubility of tantalum in Ta₂C. The core is made up of Ta₂C precipitated primarily at the grain boundaries of a tantalum matrix. A decreasing solubility of carbon in tantalum results in this. The structure described above for Figure 4 may be better understood from Figure 6. Here a 200X photomicrograph of the radial structure of sample 25AA is shown along with a plot of the radial carbon-tantalum gradient. This gradient was drawn from the solubility limits shown at 2750°C and room temperature in the phase diagram of Figure 5* as well as from the microstructure and boundary location of structurally dissimilar areas. The following can be deduced: at the end of the calibration run TaC occupied the radial area from 0 to 0.004", and was followed by Ta₂C from 0.004" to 0.013", while tantalum with carbon in solution, made up the core. Upon cooling, the decreased solubility of tantalum in TaC resulted in Ta₂C precipitation in a TaC matrix between 0.002" and 0.004". The mole ratio of tantalum to carbon in this region must, therefore, have been between the matrix for stable TaC at 2750°C, i. e., $\frac{N.Ta}{N.C.} = 1.45$, and the maximum for stable TaC at room temperature, i. e., $\frac{N.Ta}{N.C.} = 1.43$. Precipitation does not occur between 0 and 0.002" implying that $\frac{N.Ta}{N.C.} \leq 1.43$ exists here. (The gradient line for this area is drawn dotted since in actuality it might be curved.) Also, between 0.009" and 0.013" Ta is rejected into a Ta₂C matrix. Here the mole ratio must have been between the maximum for stable Ta₂C at 2750°C, 2.13, and the maximum for room-temperature stable Ta₂C, 2.09. In the 0.004" to 0.009" area a ratio less than 2.09 existed since precipitation did not occur. This area is limited to a maximum mole ratio of 2.00, however, since Ta₂C does not exist at higher values. Rejection of carbon as grain

* The accuracy of the phase diagram of Figure 5 is questionable. It was composed from the data given in references 1 and 2 which disagree somewhat in solubility limits, phase field shapes and eutectic temperatures. This, however, is of no consequence since the diagram is used only as an academic tool to aid the explanation of the microstructure developed in the code 25 wire.

boundary Ta_2C occurs in the once tantalum (with carbon in solution) core because the maximum carbon-tantalum mole ratio at $2750^{\circ}C$, 0.03, is much higher than the room temperature ratio of approximately zero.

The wire core, Ta , would tend to melt at temperatures slightly above the $2800^{\circ}C$ eutectic. This behavior is exemplified by the wire cross-sectional photomicrograph in Figure 7. Shown here is sample 25EE which was heated at 2843° to $2853^{\circ}C$. Core melting, characterized by the dendritic structure, has occurred. The layers surrounding the melted core are as described above and remain solid.

The Ta core would disappear in time for a sample held at any temperature at which carbon diffusion will measurably occur, due to growth of the TaC and Ta_2C phases. At even longer times the Ta_2C phase would disappear resulting in TaC only. The calibration time was, in general, not sufficient to eliminate the above mentioned inner layers. However, samples run at temperatures much higher than the Ta core melting point did reveal some completely carburized structure. An example of this is given in Figure 8. This photograph shows a cross section through sample 25EE, the same specimen represented in Figure 7. The Figure 8 photograph was taken somewhat away from the area represented in Figure 7, and shows the elimination of the central, once-molten, core through growth of the Ta_2C phase. The once-molten area shown in Figure 7, therefore, was being reduced through carbide growth, and probably would have disappeared in a few more minutes at temperature. Therefore, specimens heated to temperature higher than the solid solution carbon-metal core melting point may not show any dendritic signs of melting if time or temperature are great enough for carbide growth to eliminate its evidence.

As mentioned above, the five minute calibration time at temperature was, in general, not long enough to allow carbide growth to extend to the center of the wire. Reactor operation, however, could provide time or temperature sufficient for complete carburization to occur, thus eliminating even metallography as a method for determining if melting has occurred. In the NRX-A2 test it is probable that temperatures in the area of codes 19, 21, 22, 24, and 25 will not be attained since

it is not a full power run. In addition to this, the time of the run (4-5 minutes) will probably not be long enough for complete carburization to be a problem. In future, longer time full power tests, however, this problem could be a real one, and different temperature measuring procedures or materials may have to be used.

Each of the codes 19, 21, 22, and 24 underwent carburization phenomena similar to that described above for pure tantalum. This is easily understood since they consist of tantalum-niobium alloys, niobium being almost identical chemically to tantalum. High-temperature surface carbide layers developed on these materials during calibration and evidence of melting was restricted to the interior wires. The phenomena mentioned above of complete carburization and elimination of the once-melted central core occurred in only one sample of all those codes examined. A tendency toward complete carburization, however, could generally be noted with increasing calibration temperature per given code.

The data generated for codes 19, 21, 22, 24, and 25 are entered in tables of the same number. A glance at these results reveals the inconsistent melting patterns achieved with judgment by the tip geometry technique. It was first believed that these discrepancies were due to intermixing of wire codes, but chemical analysis performed on used, as well as unused samples, showed no mix-up to exist. Metallographic preparation of a few samples pointed out the above mentioned carburization phenomena, and it was decided that these wires should be calibrated metallographically. To do this, several samples from each code representing a large calibration temperature range were prepared identically to the sample shown in Figure 4. The specimens were then microscopically observed for signs of melting as depicted by the occurrence of dendritic (once melted) internal regions. Fortunately, a dendritic structure is very easily discovered metallographically. Figure 9 gives representative examples of this structure observed in two internally melted wires. In Figure 10 100X photographs of point microstructure and 200X photographs representing radial structure at a position somewhat away from the point are shown for several code

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19 samples. Point microstructures are included to reveal the retention of tip geometry even when core melting occurs. Structures shown represent those formed in the temperature range, 2317° to 2433°C . Samples 19AA, 19BB, and 19CC do not show any evidence of a once-melted center. Samples 19F, 19D, 19DD, and 19EE all show definitely once-melted interiors. Melting occurs between 2381° and 2398°C or approximately 2390°C for this code. Carbide outer layers are quite evident in these samples, and vary somewhat in thickness. This variability is probably due to increased carbon diffusion with increased temperature, and the inconsistency in the time taken to achieve exactly the calibration temperature, which may have been from a few seconds to about five minutes on a given sample.

The structures on code 21 samples, Figure 11, show essentially the same pattern. Samples 21AA and 21BB representing the calibration range of 2528° to 2555°C do not show a structure characteristic of internal melting. Samples 21CC, 21B, 21DD, and 21EE all reveal some degree of internal melting. The transition appears to occur between 2555° and 2576°C or 2565°C as an average of the code's melting point. The carbide surface layers show a definite increase in thickness with increase in calibration temperature and their depth is generally greater as compared with the code 19 samples.

Code 22 photographs shown in Figure 12 reveal solid to liquid transition somewhere between 2625° and 2632°C . Both samples 22CC and 22A were run essentially over this temperature range, 22A indicating some core melting while 22CC reveals no melting. Two samples run below this temperature range were unmelted while five samples run above it contained internal dendritic structures (only two are shown). The melting temperature for code 22 is, therefore, in the vicinity of 2630°C . Carbide thickness attained on these samples is equivalent to or slightly greater than that developed on code 21 samples.

Codes 24 and 25 indicate melting temperatures of 2710° and 2790°C , respectively. The sample photographs upon which this conclusion is based are

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given in Figures 13 and 14. The melting temperatures were deduced in a manner identical to that used for codes 19, 21, and 22 and will not be repeated here. It should be noted that the carbide layer thickness developed in these samples was quite extensive. One sample, 24EE, carburized completely and revealed absolutely no evidence of a dendritic core throughout its entire length although the core must have been molten at one time since the wire was heated well above the metal-carbon solid solution core melting point. Also, sample 25EE (not shown in Figure 14) showed areas of complete carburization as well as dendritic central core regions (refer to discussion of Figures 7 and 8).

The metallographic data imply that codes 24 and 25 will tend to completely carburize at temperatures in the vicinity of their melting points if held there for more than about five minutes. Because of this they cannot be considered reliable for full power tests in excess of five minutes. The rate of carbide formation on codes 19, 21, and 22 at temperatures in the vicinity of their melting points was, however, much slower. It is estimated that these codes are sufficient for 10 minute full power tests.

It is possible that under reactor environmental conditions local core temperatures exceeding the melting temperature of one or more of codes 19, 21, 22, 24, and 25 will be achieved in a matter of a few seconds. If this is true, the carbide surface layers may be too thin and, therefore, correspondingly weak enough to collapse under the pressure generated by the internal liquid.* However, it should be remembered that temperatures within about 50°C of the calibration temperature were attained within a few seconds, and samples wherein internal melting did occur still developed enough surface carbide to resist deformation.

* Contact with LASL personnel revealed that they encountered the same carburization difficulties when "slowly" achieving calibration temperatures. They were able to avoid the problem, however, by rapidly attaining temperature thereby minimizing the surface carbide thickness, and enhancing the chance for internal melting to rupture the carbide and provide visual evidence of melting.

Occasionally, as noted in the data on these codes, the carbide layers did collapse at the region of the wire tip allowing melting to be detected visually. This occurred randomly under our calibration procedure, but may be induced by reactor conditions of rapid temperature transients, vibration, etc., thereby enabling radiographic temperature determination to be used. In addition to this, large internal voids may be used as a radiographic indication that melting has occurred. Porosity occurred randomly in capsules heated above their melting temperature occasionally occurring in code 19, 21, and 22 samples, and occurring extensively in code 24 and 25 samples. Some of the photographs of Figures 10 through 14 show large voids which have developed at the point areas of some wires. Voids were also observed along the body of melted wires. However, because porosity did not develop in all samples which showed internal melting, its absence should not be used as an indication that melting did not occur.

If radiography leaves some doubt as to the temperature of a given region, and it appears it may be in the vicinity of the indicating temperature of these codes, hot cell metallography will have to be performed. In this case micro-structural examination of the wire core will have to be used to determine whether melting has or has not occurred. If metallography must be used, and it uncovers a completely carburized structure, i. e., Figure 8 and sample 24EE in Figure 13, one cannot be sure of the temperature attained at the wire location since this may occur with sufficient time at temperatures below the wire core melting point. It is doubtful that this will occur in NRX-A2 because of the low-power, short time schedule. However, as mentioned before, longer time full power tests, i. e., A3, A4, etc., could provide conditions under which this may develop.

CONCLUSIONS

- 1) All WANL thermal capsule wires revealed visual or metallographic indications of melting within approximately $\pm 50^{\circ}\text{C}$ of their predicted temperatures.
- 2) Codes numbered 1 through 18, 20, 23, 26, and 27 readily deform when heated in the vicinity of their melting points. Radiography will probably provide a simple method for detecting melting in these codes.
- 3) Codes 19, 21, 22, 24, and 25 form surface carbides which resist deformation even when melting occurs at the lower-carbon, interior section of the wires. Metallographic detection of melting may, therefore, be necessary if core temperatures are suspected to be in the area of the melting points of these codes and radiography does not detect any indications of melting.
- 4) A tendency to form large shrinkage cavities when cooled was observed in internally melted samples of codes 19, 21, 22, 24, and 25. This was more prevalent in codes 24 and 25, but occurred on occasion in codes 19, 21, and 22. Radiographic detection of gross porosity should be interpreted as a sign that melting has occurred even if the wire point has not deformed. The absence of such porosity, however, is not an indication that melting did not occur, and metallographic examination for melting should be used if doubt exists.
- 5) Carbide growth to the center of the wire could eliminate any metallographic evidence of melting in codes 19, 21, 22, 24, and 25. This occurred in only a few samples tested under the calibration procedure which involved approximately 5 to 10 minutes at elevated temperatures. The procedure involved a few seconds to attain a temperature within about 50°C of the calibration temperature, followed by a few more seconds to a maximum of five minutes cautiously approaching this temperature. The samples were

then held at temperature for exactly 5 more minutes. It is estimated that codes 19, 21, and 22 can be operated for about 10 minutes at temperatures in the vicinity of their melting points without completely carburizing. Codes 24 and 25, however, tend to completely carburize at times longer than about 5 minutes at temperatures around their melting points and, therefore, can only be considered reliable for 5 minute tests.

RECOMMENDATIONS

- 1) Future work should be done to uncover new material combinations or temperature indicating techniques for measurements above 2300°C . It is possible that various metal oxide or metal oxide-graphite combinations may provide systems wherein melting could be easily detected radiographically. In addition to this it may be possible to substitute one metal-graphite combination for several combinations by using a measurement of the thickness of carbide case developed during a reactor run as an indication of the temperature attained. For example, pure tantalum could probably be used for the range 2000° to 2800°C . Plots of carbide thickness vs. time for specific temperatures in this range, along with the carbide thickness attained during the time of a specific reactor run, would then allow one to deduce a temperature. This, of course, assumes that steady-state core temperatures are achieved in a matter of a few seconds and hold constant for the duration of the test. The technique might prove useful for reactor tests wherein the time occupied by transient, start-up, temperature conditions is short compared to the time under steady-state conditions, i. e., long time reactor tests.
- 2) Consideration should be given to the possibility of increasing the wire diameter used on codes 19, 21, 22, 24, and 25. This would reduce the possibility of complete carburization as well as allow the formation of a larger volume of liquid core to develop in those wires which attain temperatures higher than their melting points. A greater liquid volume may aid in deforming or rupturing the carbide case and in the development of shrinkage cavities upon cooling, a

phenomenon which would promote radiographic melting detection.

- 3) The need for metallographic facilities should be anticipated for post-mortem examination of codes 19, 21, 22, 24, and 25 if radiography proves to be insufficient.

REFERENCES

- 1) Defense Metals Information Center, Report 152, April 28, 1961.
- 2) Storms, E. K., "A Critical Review of Refractories, " Part I, Selected Properties of Group 4a, 5a, and 6a Carbides, LAMS-2674, March 15, 1962.

ACKNOWLEDGEMENTS

The cooperation of the Los Alamos Scientific Laboratory in discussing their thermal capsule calibration experience with WANL personnel, and the loan of their calibration equipment is appreciated.

TABLE 1

Calibration Results for WANL Wire Code Number 1*

Pure Aluminum
Batch No. - Unknown
Group No. - 978DO74G24E

<u>Calibration Temperature (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
655 °C	Pointed	1
660 °C	Rounded	2
678-698 °C	Rounded	3
678-698 °C	Rounded	4

Melting Temperature - 660 °C

*Calibration runs were performed in an electric resistance furnace under protection of argon. The capsule lid was retained on the sample, and temperature was measured by a thermocouple wrapped around the sample.

TABLE 2

Calibration Results for WANL Wire Code Number 2*

72 w/o Ag - 28 w/o Cu
Batch No. - 1792
Group No. - 978DO74G11E

<u>Calibration Temperature (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
774	Pointed	1
774	Pointed	2
780	Rounded	3
787	Rounded	4

Melting Temperature - 775 °C

*Calibration runs were performed in an electric resistance furnace under protection of argon. The capsule lid was retained on the sample, and temperature was measured by a thermocouple wrapped around the sample.

TABLE 3

Calibration Results for WANL Wire Code Number 3

80 w/o Au - 20 w/o Cu
 Batch No. - E.I. 4606
 Group No. - 978DO74GO7E

<u>Calibration Temperature (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
876-884	Pointed	1
892-897	Pointed	2
908-914	Pointed	3
902-917	Rounded	4
923-928	Rounded	5

Melting Temperature - 915 °C

TABLE 4

Calibration Results for WANL Wire Code Number 4

Pure Silver
Batch No. - E. I. 2006
Group No. - 978DO74G25E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
875-879	Pointed	1
952-958	Pointed	2
966-977	Rounded	3
990-1000	Rounded	4
1006-1011	Rounded	5

Melting Temperature - 970 °C

TABLE 5

Calibration Results for WANL Wire Code Number 5

82 w/o Ag - 18 w/o Pd

Batch No. - 13052

Group No. - 978DO74G05E

Calibration Temperature Range (°C)	Wire Tip Geometry	Photograph Number
1009-1022	Pointed	--
1062-1077	Pointed	1
1083-1090	Slightly Rounded	2
1090-1098	Rounded	3
1097-1107	Rounded	4
1098-1108	Rounded	5

Melting Temperature - 1095 °C

TABLE 6

Calibration Results for WANL Wire Code Number 6

67 w/o Ag - 33 w/o Pd

Batch No. - 14951

Group No. - 978DO74G13E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
1152-1157	Pointed	--
1192-1200	Pointed	1
1192-1203	Pointed	2
1203-1213	Pointed	3
1217-1222	Rounded	4
1416-1223	Rounded	5

Melting Temperature - 1220 °C

TABLE 7

Calibration Results for WANL Wire Code Number 7

50 w/o Ag - 50 w/o Pd

Batch No. - 1453

Group No. - 978DO74G16E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
1242-1260	Pointed	1
1285-1294	Slightly Rounded	2
1300-1303	Slightly Rounded	3
1313-1320	Rounded	4
1313-1330	Rounded	5

Melting Temperature - 1300 °C

TABLE 8

Calibration Results for WANL Wire Code Number 8

75 w/o Pd - 25 w/o Ag
Batch No. - E 1349
Group No. - 978DO74G10E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
1390-1396	Pointed	1
1410-1417	Slightly Rounded	2
1407-1418	Slightly Rounded	3
1407-1418	Slightly Rounded	4
1432-1439	Rounded	5

Melting Temperature - 1420 °C*

*Because of the similar and almost melted tip characteristics of samples 2 through 4, they were considered representative of the melting temperature of this wire.

TABLE 9

Calibration Results for WANL Wire Code Number 9

85 w/o Pd - 15 w/o Ag
Batch No. - 1511
Group No. - 978DO74G04E

Calibration Temperature Range (°C)	Wire Tip Geometry	Photograph Number
1400-1410	Pointed	1
1445-1450	Pointed	2
1455-1465	Rounded	3
1463-1468	Rounded	4
1468-1476	Rounded	5

Melting Temperature - 1460 °C

TABLE 10

Calibration Results for WANL Wire Code Number 10

Pure Palladium
Batch No. - E. I. 6007
Group No. - 978DO74G17E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
1472-1481	Pointed	1
1484-1489	Pointed	2
*1484-1489	Pointed	--
*1495-1500	Rounded	--
1494-1503	Rounded	3
1500-1505	Rounded	4
1512-1522	Rounded	5

Melting Temperature - 1495 °C

*Run after completion of calibration on all wires as an operator and pyrometer check.

TABLE 11

Calibration Results for WANL Wire Code Number 11

90 w/o Pd - 10 w/o Ir

Batch No. - 11486

Group No. - 978DO74G01E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
1465-1490	Pointed	1
1485-1487	Pointed	2
1500-1502	Rounded	3
1515-1517	Rounded	4
1530-1535	Rounded	5
1540-1550	Rounded	--

Melting Temperature - 1495 °C

TABLE 12

Calibration Results for WANL Wire Code Number 12

Pure Rhodium
Batch No. - 7005
Group No. - 978DO74G18E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
1627-1639	Pointed	1
1632-1650	Pointed	2
1657-1667	Pointed	3
1670-1680	Rounded	4
1675-1686	Rounded	5
1712-1720	Rounded	6

Melting Temperature - 1675 °C

TABLE 13

Calibration Results for WANL Wire Code Number 13

88 w/o Pt - 12 w/o Ir

Batch No. - 14142

Group No. - 978DO74G03E

Calibration Temperature Range (°C)	Wire Tip Geometry	Photograph Number
1756-1761	Pointed	1
1758-1761	Pointed	2
1772-1774	Rounded	3
*1782-1792	Pointed	4
1782-1796	Rounded	5
1812-1822	Rounded	6

Melting Temperature - 1770 °C

*Considered an erroneous temperature reading.

TABLE 14

Calibration Results for WANL Wire Code Number 14

50 w/o Ir - 50 w/o Rh

Batch No. - 5959

Group No. - 978D074G15E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
1878-1889	Pointed	1
1878-1891	Pointed	2
1895-1900	Rounded	3
1900-1910	Slightly Rounded	4
1913-1922	Rounded	5
1922-1928	Rounded	6

Melting Temperature - 1895 °C

TABLE 15

Calibration Results for WANL Wire Code Number 15

60 w/o Ir - 40 w/o Rh

Batch No. - 5942

Group No. - 978DO74G14E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
1977-1993	Pointed	1
1977-1993	Pointed	2
1986-1997	Slightly Rounded	3
2010-2020	Rounded	4

Melting Temperature - 2010 °C

TABLE 16

Calibration Results for WANL Wire Code Number 16

80 w/o Ir - 20 w/o Rh

Batch No. - 12302

Group No. - 978DO74G06E

Calibration Temperature Range (°C)	Wire Tip Geometry	Photograph Number
2115-2126	Pointed	1
2142-2149	Pointed	2
2148-2152	Rounded	3
2153-2163	Rounded	4
2170-2175	Rounded	5

Melting Temperature - 2150 °C

TABLE 17

Calibration Results for WANL Wire Code Number 17

Pure Molybdenum
Batch No. - P. M. 523-S-2
Group No. - 978DO74G20E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
2175-2192	Pointed	1
2178-2187	Pointed	2
2187-2198	Rounded	3
*2198-2208	Pointed	4
2220-2231	Rounded	5

Melting Temperature - 2195 °C

*Considered an erroneous temperature reading.

TABLE 18

Calibration Results for WANL Wire Code Number 18

Pure Iridium
Batch No. - E. I. 4002
Group No. - 978DO74G19E

Calibration Temperature Range (°C)	Wire Tip Geometry	Photograph Number
2254-2286	Pointed	1
*2275-2285	Pointed	--
*2278-2287	Pointed	--
2277-2297	Pointed	2
2298-2310	Rounded	3
2310-2318	Rounded	4
*2309-2320	Rounded	--
2312-2330	Rounded	5
*2320-2330	Rounded	--

Melting Temperature - 2305 °C

*Run after completion of calibration on all wires as an operator and pyrometer check.

TABLE 19

Calibration Results for WANL Wire Code Number 19

79 w/o Nb - 21 w/o Ta

Batch No. - Unknown

Group No. - 978DO74G08E

Sample Number	Calibration Temperature Range (°C)	Wire Tip ⁽²⁾ Geometry	Photomicrograph Number	Microstructural Comments ⁽³⁾
19AA	2312-2317	Pointed	5129	B
19BB	2350-2355	Pointed	5130	B
19CC	2375-2381	Pointed	5131	B
19A	2375-2386	Pointed	--	--
19F	2386-2398 ⁽¹⁾	Pointed	5082	A
19D	2399-2410	Pointed	5081	A
19B	2404-2410	Pointed	(4)	--
19DD	2410-2415	Pointed	5132	A
19G	2410-2420	Pointed	--	--
19C	2420-2431	Rounded	--	--
19E	2425-2431	Slightly rounded	(4)	--
19EE	2428-2433	Rounded	5133	A
19M	2446-2461	Rounded	--	--
19J	2442-2460	Pointed	--	--
19J re-ran	2471-2482	Pointed	--	--
19J re-ran	2500-2507	Pointed	--	--
19K	2448-2458	Pointed	(4)	--
19L	2453-2464	Rounded	--	--
19H	2453-2464	Pointed	--	A

Melting Temperature - 2390 °C

(1) Held at temperature for ten minutes.

(2) This wire was calibrated metallographically because of the inconsistent relationship between temperature and wire tip geometry. Refer to Figure 10 for these results.

(3) A: sample shows signs of internal melting.

B: sample does not show any sign of internal melting.

(4) These samples were chemically analyzed. Results showed proper composition.

TABLE 20

Calibration Results for WANL Wire Code Number 20

Pure Rhenium
Batch No. - R. W. E. 12
Group No. - 978DO74G22E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
2452-2463	Pointed	1
2438-2463	Pointed	2
*2476-2488	Pointed	3
*2476-2481	Rounded	4
2498-2508	Rounded	5
2498-2510	Rounded	6
2510-2525	Rounded	7

Melting Temperature - 2480 °C*

*Because of the overlap of temperature range and the result of one sample melting while the other did not melt, the true melting point was considered around 2480 °C.

TABLE 21

Calibration Results for WANL Wire Code Number 21

 33 w/o Nb - 67 w/o Ta
 Batch No. - Unknown
 Group No. - 978DO74G12E

Sample Number	Calibration Temperature Range (°C)	Wire Tip ⁽¹⁾ Geometry	Photomicrograph Number	Microstructural Comments ⁽²⁾
21A	2520-2530	Pointed	--	--
21AA	2525-2528	Pointed	5134	B
21BB	2550-2555	Pointed	5135	B
21CC	2570-2576	Pointed	5136	A
21B	2581-2590	Pointed	5079	A
21DD	2586-2596	Pointed	5137	A
21M	2608-2613	Pointed	--	--
21C	2608-2619	Pointed	--	--
21EE	2620-2625	Pointed	5138	A
21E	2619-2636	Slightly Rounded	--	--
21G	2633-2643	Pointed	--	--
21G re-run	2903-2908	Rounded	--	--
21F	2633-2643	Rounded	--	--
21K	2642-2652	Pointed	--	--
21I	2642-2652	Pointed	--	--
21L	2642-2652	Rounded	--	--
21D	2650-2655	Rounded	(3)	--
21H	2658-2670	Pointed	3978	A
21J	2665-2674	Pointed	--	--
21J re-run	2642-2652	Pointed	--	--
21J re-run	2686-2697	Pointed	--	--
21J re-run	2702-2713	Pointed	--	--
21J re-run	2720-2730	Pointed	--	--
21J re-run	2753-2763	Pointed	(3)	--

Melting Temperature - 2565 °C

(1) This wire was calibrated metallographically because of the inconsistent relationship between temperature and wire tip geometry. Refer to Figure 11 for these results.

(2) A: sample shows signs of internal melting.
 B: sample does not show any sign of internal melting.

(3) These samples were chemically analyzed. Results show proper composition.

TABLE 22

Calibration Results for WANL Wire Code Number 22

22 w/o Nb - 78 w/o Ta
Batch No. - Unknown
Group No. - 978DO74G09E

Sample Number	Calibration Temperature Range (°C)	Wire Tip ⁽¹⁾ Geometry	Photomicrograph Number	Microstructural Comments ⁽²⁾
22AA	2559-2564	Pointed	5139	B
22BB	2595-2600	Pointed	5140	B
22A	2625-2631	Slightly Rounded	3972	A
22CC	2626-2632	Pointed	5141	B
22D	2631-2653	Pointed	--	--
22G	2631-2674	Pointed	--	--
22C	2642-2653	Pointed	5431	A
22B	2648-2658	Rounded	--	--
22E	2663-2674	Slightly Rounded	--	A
22F	2663-2674	Slightly Rounded	--	A
22DD	2663-2675	Pointed	5142	A
22H	2686-2697	Slightly Rounded	--	--
22E	2708-2713	Slightly Rounded	--	A
22I	2708-2742	Rounded	--	--

Melting Temperature - 2630 °C

(1) This wire was calibrated metallographically because of the inconsistent relationship between temperature and wire tip geometry. Refer to Figure 12 for these results.

(2) A: sample shows signs of internal melting.
B: sample does not show any sign of internal melting.

TABLE 23

Calibration Results for WANL Wire Code Number 23

Pure Tungsten
Batch No. - Lot 23
Group No. - 978DO74G23E

<u>Sample Number</u>	<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	
23C	2674-2686	Pointed	<p>Ran with thermal capsule cover off.</p>
23A	2691-2708	Rounded*	
23B	2708-2713	Slightly Rounded	
23D	2713-2720	Pointed	
23E	2731-2736	Rounded*	
23H	2663-2674	Pointed	<p>Ran with thermal capsule cover on to prevent erroneous temperature readings due to expansion upon melting.</p>
23I	2695-2698	Pointed	
23G	2698-2708	Rounded	
23J	2708-2720	Slightly Rounded	
23F	2731-2748	Rounded	

Melting Temperature - 2705 °C

*These samples melted and expanded approximately one-quarter inch out of the graphite case and into the cassette. A lowering of brightness (temperature) occurred due to this. Temperature readings were not taken prior to expansion and, thus, this data represents lower temperatures than that at which melting occurred.

TABLE 24
Calibration Results for WANL Wire Code Number 24

10 w/o Nb - 90 w/o Ta
 Batch No. - Unknown
 Group No. - 978DO74G02E

<u>Sample Number</u>	<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip⁽¹⁾ Geometry</u>	<u>Photomicrograph Number</u>	<u>Microstructural Comments⁽²⁾</u>
24AA	2688-2693	Pointed	5144	B
24FF	2698-2703	Pointed	5434	B
24GG	2715-2720	Pointed	5435	A
24BB	2732-2738	Pointed	5145	A
24CC	2742-2749	Pointed	5146	A
24A	2746-2753	Pointed	--	--
24DD	2763-2768	Pointed	5147	--
24B	2776-2781	Pointed	--	--
24C	2802-2807	Pointed	--	--
24EE	2803-2808	Slightly Rounded	5148	A
24G	2802-2810	Pointed	--	--
24E	2812-2830	Slightly Rounded	--	--
24I	2820-2830	Pointed	--	--
24J	2841-2846	Rounded	--	--
24D	2841-2852	Rounded	--	--
24H	2846-2852	Rounded	--	--

Melting Temperature - 2710 °C

(1) This wire was calibrated metallographically because of the inconsistent relationship between temperature and wire tip geometry. Refer to Figure 13 for these results.

(2) A: sample shows signs of internal melting.

B: sample does not show any sign of internal melting.

TABLE 25

Calibration Results for WANL Wire Code Number 25

Pure Tantalum
Batch No. - M. G.-63
Group No. - 978DO74G21E

Sample Number	Calibration Temperature Range (°C)	Wire Tip ⁽¹⁾ Geometry	Photomicrograph Number	Microstructural Comments ⁽²⁾
25AA	2747-2749	Pointed	5149	B
25BB	2778-2788	Pointed	5150	B
25KK	2783-2788	Pointed	5439	A
25LL	2793-2798	Pointed	5440	A
25CC	2798-2806	Pointed	5151	A
25DD	2818-2830	Pointed	5152	A
25II	2820-2830	Pointed	--	--
25JJ	2820-2843	Pointed	--	--
25B	2822-2830	Pointed	--	--
25C	2830-2835	Pointed	--	--
25H	2830-2835	Pointed	--	--
25D	2842-2852	Rounded	--	--
25EE	2843-2853	Pointed	--	A
25A	2846-2862	Rounded	--	--
25E	2858-2863	Pointed	--	--
25I	2863-2869	Rounded	--	--
25F	2875-2885	Pointed	--	--
25FF	2875-2885	Pointed	--	A
25GG	2885-2895	Rounded	--	--
25G	2903-2908	Rounded	--	--

Melting Temperature - 2790 °C

(1) This wire was calibrated metallographically because of the inconsistent relationship between temperature and tip geometry. Refer to Figure 14 for these results.

(2) A: sample shows signs of internal melting.
B: sample does not show any sign of internal melting.

TABLE 26

Calibration Results for WANL Code Number 26

50 w/o Pt - 50 w/o Pd

Batch No. 6353

Group No. - 978DO74G26E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
1565-1570	Pointed	1
1615-1620	Pointed	2
1613-1622	Slightly Rounded	3
1622-1630	Rounded	4
1650-1653	Rounded	5

Melting Temperature - 1625 °C

TABLE 27

Calibration Results for WANL Wire Code Number 27

85 w/o Pt - 15 w/o Cu
Batch No. - 14969
Group No. - 978DO74G27E

<u>Calibration Temperature Range (°C)</u>	<u>Wire Tip Geometry</u>	<u>Photograph Number</u>
1535-1555	Pointed	1
1565-1580	Pointed	2
1590-1595	Rounded	3
1595-1605	Rounded	4
1625-1630	Rounded	5

Melting Temperature - 1590 °C

TABLE 28

WANL Thermal Capsule Code Numbers, Composition,
Predicted Melting Temperature, and Calibration Temperature

Code Number	Composition (w/o)	Predicted Temperature (°C)	Calibration Temperature (± 30 °C)
1	Pure Aluminum	660	660
2	72 Ag - 28 Cu	779	775
3	80 Au - 20 Cu	890	915
4	Pure Silver	960	970
5	82 Ag - 18 Pd	1060	1095
6	67 Ag - 33 Pd	1170	1220
7	50 Ag - 50 Pd	1280	1300
8	75 Pd - 25 Ag	1395	1420
9	85 Pd - 15 Ag	1450	1460
10	Pure Palladium	1530	1495
11	90 Pd - 10 Ir	1510	1495
12	Pure Rhodium	1700	1675
13	88 Pt - 12 Ir	1800	1770
14	50 Ir - 50 Rh	1900	1895
15	60 Ir - 40 Rh	2000	2010
16	80 Ir - 20 Rh	2120	2150
17	Pure Molybdenum	2180	2195
18	Pure Iridium	2300	2305
19	79 Nb - 21 Ta	2375	2390*
20	Pure Rhenium	2450	2480
21	33 Nb - 67 Ta	2570	2565*
22	22 Nb - 78 Ta	2630	2630*
23	Pure Tungsten	2700	2705
24	10 Nb - 90 Ta	2750	2710*
25	Pure Tantalum	2850	2790*
26	50 Pt - 50 Pd	1600	1625
27	85 Pt - 15 Cu	1620	1590

*Calibrated by metallographically observing evidence of internal melting.

FIGURE 1. THERMAL CAPSULE AND CASSETTE CONSTRUCTION 1X

TOP TO BOTTOM
 DISSASSEMBLED GRAPHITE
 CASSETTE ARRANGED IN
 ORDER OF ASSEMBLY AND
 INCLUDING A THERMAL
 CAPSULE

TOP TO BOTTOM
 ASSEMBLED GRAPHITE CASSETTE
 FOLLOWED BY A DISSASSEMBLED
 THERMAL CAPSULE SHOWING
 GRAPHITE CASE AND CAP AND
 POINTED METAL WIRE

FIGURE 2. CLOSE-UP PHOTOGRAPH OF HEATING UNIT.
PHOTOGRAPH TAKEN OBLIQUELY FROM SIDE OF INDUCTION
HEATING UNIT SHOWING EXTERIOR TAPE-WRAPPED INDUCTION
COIL, QUARTZ TUBE AND INTERIOR COPPER CONCENTRATOR
SURROUNDING A GLOWING CASSETTE.

FIGURE 3. PHOTOGRAPH OF CALIBRATION EQUIPMENT.

OPERATOR TAKING A THERMAL CAPSULE CALIBRATION TEMPERATURE READING.
 CENTER: LEEDS & NORTHRUP OPTICAL PYROMETER. LEFT OF CENTER: INDUCTION
 COIL SURROUNDING QUARTZ TUBE WITH PRISM ON GLASS PORT OF TOP FLANGE.
 REAR: INDUCTION UNIT . FAR LEFT: HELIUM SUPPLY BOTTOM: ROUGH PUMP.

TaC

TaC WITH PRECIPITATED Ta₂C

Ta₂C

Ta₂C WITH PRECIPITATED (Ta)

(Ta) WITH PRECIPITATED Ta₂C

FIGURE 4. MICROSTRUCTURAL CROSS-SECTION THROUGH SAMPLE 25AA. SAMPLE WAS PURE TANTALUM HEATED TO 2747 - 2749°C FOR 5 MINUTES

20CC H₂O - 50CC HNO₃ - 30gms NH₄F HF ETCH

FIGURE 5. PHASE DIAGRAM OF THE TANTALUM - CARBON SYSTEM (1, 2)

MIRCO 5149

200X

20CC. H_2O - 50CC. HNO_3 - 30gms NH_4F ·HF ETCH

FIGURE 6 RADIAL CARBON AND TANTALUM GRADIENT IN SAMPLE 25AA. THIS SPECIMEN WAS HEATED TO 2747 -2749 °C HELD FOR APPROXIMATELY 5 MINUTES AND RAPIDLY COOLED. WHILE AT TEMPERATURE REGIONS 1 AND 2 CONSISTED OF TaC , 3 AND 4, OF Ta_2C AND 5 OF TANTALUM WITH CARBON IN SOLID SOLUTION. WHEN COOLED TO ROOM TEMPERATURE THE TANTALUM CONTENT WAS GREATER THAN THE SOLUBILITY LIMIT IN TaC IN REGION 2 (1.43 N.Ta/N.C.) AND Ta_2C PRECIPITATED. IN REGION 4 THE SOLUBILITY OF Ta IN Ta_2C (2.09² N.Ta/N.C.) WAS EXCEEDED RESULTING IN (Ta) PRECIPITATION, WHILE PRECIPITATION OF Ta_2C OCCURRED IN REGION 5 BECAUSE OF THE EXTREMELY LOW ROOM TEMPERATURE SOLUBILITY OF CARBON IN TANTALUM.

FIGURE 7. CROSS SECTIONAL STRUCTURE THROUGH TANTALUM THERMAL CAPSULE WIRE SHOWING ONCE MOLTEN CORE. WIRE WAS HEATED IN THE RANGE 2843 TO 2853 °C FOR 5 MINUTES. HEATING ABOVE THE EUTECTIC TEMPERATURE RESULTED IN INTERNAL MELTING.

SAMPLE 25EE
MICRO 5153
100X

TaC ———→
 $\text{TaC WITH PRECIPITATED } \text{Ta}_2\text{C}$ ———→
 Ta_2C ———→
 $\text{Ta}_2\text{C WITH PRECIPITATED (Ta)}$ ———→

FIGURE 8. CROSS SECTIONAL STRUCTURE THROUGH TANTALUM THERMAL CAPSULE WIRE SHOWING COMPLETELY CARBURIZED STRUCTURE. WIRE WAS HEATED IN THE RANGE 2843 TO 2853 °C FOR 5 MINUTES. IN THIS SECTION, GROWTH OF Ta_2C COMPLETELY ELIMINATES THE MOLTEN CENTRAL CORE. IN OTHER SECTIONS OF THIS SAMPLE EVIDENCE OF A ONCE - MOLTEN CENTER REGION IS STILL PRESENT (REFER TO FIGURE 7.)

SAMPLE NO. 21H MICRO 3978

250X

SAMPLE NO. 25DD MICRO 5132

250X

20CC.H₂O - 50CC.HNO₃ -30gms. NH₄F. HF ETCH.

FIGURE 9 MICROSTRUCTURE CHARACTERISTIC OF ONCE
MELTED AREAS IN CODES 19, 21, 22, 24, & 25

FIGURE 10. POINT AND RADIAL BODY MICROSTRUCTURES FOR VARIOUS CODE 19 SAMPLES (21 w/o Ta - 79 w/o Nb)
SOLID TO LIQUID TRANSITION OCCURS BETWEEN 2381 AND 2398 °C, SAMPLES 19CC AND 19F.

20 CC H₂O - 50 CC HNO₃ - 30gms NH₄F·HF ETCH

FIGURE 11 POINT AND RADIAL BODY MICROSTRUCTURES FOR VARIOUS CODE 21 SAMPLES
(67 w/o Ta - 33 w/o Nb) SOLID TO LIQUID TRANSITION OCCURS BETWEEN 2555 AND 2576 °C

20CC. H₂O - 50CC. HNO₃ - 30gms NH₄F · HF ETCH

FIGURE 12. POINT AND RADIAL BODY MICROSTRUCTURE ON VARIOUS CODE 22 SAMPLES (78 w/o Ta - 22 w/o Nb)
SOLID TO LIQUID TRANSITION OCCURS AROUND 2630 °C

20CC H₂O - 50CC HNO₃ - 30gms NH₄F HF ETCH

FIGURE 13. POINT AND RADIAL BODY MICROSTRUCTURES ON VARIOUS CODE 24 SAMPLES (90 w/o Ta - 10 w/o Nb) SOLID TO LIQUID TRANSITION OCCURS BETWEEN 2703 AND 2720 °C. NOTICE THAT THE SOLID SURFACE CARBIDE LAYERS INCREASE IN DEPTH WITH INCREASING TEMPERATURE UNTIL AT AROUND 2800°C THEY EXTEND THROUGHOUT THE ENTIRE STRUCTURE WHICH ELIMINATES THE LOW CARBON-LOW MELTING CORE.

20CC. H₂O - 50CC. HNO₃ - 30gms NH₄F·HF

SAMPLE NO.25AA

MICRO 5149

SAMPLE NO.25BB

MICRO 5150

SAMPLE NO. 25KK

MICRO 5439

SAMPLE NO. 25LL

MICRO 5440

SAMPLE NO. 25CC

MICRO 5151

SAMPLE NO. 25DD

MICRO 5152

100X

100X

100X

100X

100X

100X

200X

2747 - 2749 °C

200X

2778 - 2788 °C

200X

2783 - 2788 °C

200X

2793 - 2798 °C

200X

2798 - 2806 °C

200X

2818 - 2830 °C

FIGURE 14. POINT AND RADIAL BODY MICROSTRUCTURES ON VARIOUS CODE 25 SAMPLES (PURE TANTALUM)
SOLID TO LIQUID TRANSITION OCCURS AROUND 2790°C

20CC. H₂O - 50CC. HNO₃ - 30gmc NH₄F·HF