

EXPERIMENTAL TECHNIQUES USED IN THE HIGH TEMPERATURE  
HEAT-TREATMENT OF SOME TUNGSTEN-THORIA ALLOYS

by

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December, 1964.

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SYNOPSIS

The high temperature apparatus developed in the Department of Materials for the torsional creep testing of graphite at temperatures up to 3000°C has been utilised to heat-treat tungsten-thoria alloys at temperatures up to 2500°C. Modifications to the torsional loading system have enabled some of these heat-treatments to be carried out under an applied stress.

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## Introduction

The programme of work reported here has been undertaken on behalf of Aeon Laboratories Limited, Egham, Surrey as part of their investigation into the microstructure of tungsten-thoria alloys. During the course of this investigation a need arose for the heat treatment of these materials in the temperature range 1700 - 2500°C. As a result, the Department of Materials was approached with a view to carrying out these heat treatments in the high temperature furnace at Cranfield.

The high temperature furnace was developed as part of an investigation into the creep and recovery properties of graphite at high temperature sponsored by the U.S. Department of the Army (Contract Nos. DA-91-591-EUC1759; DA-91-591-EUC2629; DA-91-591-EUC2880). The apparatus has been fully described in the reports appertaining to these contracts. Little modification was necessary to undertake the basic heat-treatments, but for the heat-treatment under stress a special gripping arrangement was required. The heat-treatment cycles and the minor modifications to the equipment are described in this report.

## Experimental

### A. Heat treatment without stress

The tungsten alloys (1 and 2% thoria) were supplied in the form of rods  $1\frac{1}{2}$  - 2 ins in length and between 0.100 and 0.125 ins diameter. The rods have been identified by cutting grooves near their ends.

The rods have been placed nearly vertically in an enclosure formed by cutting away part of the gauge length wall from a graphite torsional creep specimen as formerly used on this rig. See Fig. 1. The total radiation pyrometer was focused on the outside of the remaining part of the wall. After loading the whole chamber was evacuated, usually overnight, to a pressure of about 1 mm Hg. For the higher temperature runs the chamber was then flushed with argon, up to a pressure of 200 mm Hg and then re-evacuated. For heat-treatments at 1700°C, and for short runs at 1900°C, vacua was maintained throughout. For higher temperatures argon was admitted as the temperature was raised from

1500° to 2000°C, until at 2000°C the chamber was at atmospheric pressure. A through-flow of 5 - 10 cu. ft. per hour of argon was maintained during these tests.

The heating cycles followed a fixed pattern throughout. The power was initially switched on at a low setting; the temperature rising over about 10 minutes to about 850°C. This slow temperature rise under vacua should have enabled the surfaces to lose all adsorbed gases. A soaking period of 5 - 10 minutes was usually allowed at 850°C. The power setting was then increased, allowing the temperature to rise in 2 - 3 minutes to 1500°C, the lowest setting of the temperature controller. A soaking period of specified time, usually 15 minutes, was allowed at 1500°C.

After this soaking period the power setting was increased to the maximum possible, and using the temperature controller, the temperature was raised in uniform increments of 50°C until the required heat treatment temperature of 1700°C or 1900°C was reached. A soaking time of 4 minutes was allowed at each increment; the rise time at each increment being about 20 seconds. For the tests at 2500°C the increments were 250°C instead of 50°C, the rise time being less than 1 minute.

Soaking for a given time was followed by a relatively slow cool, the temperature being lowered in decrements of 50°C every 3 minutes down to 1500°C. From 2500°C, the decrements were 250°C. After 3 minutes at 1500°C the power setting was switched to the minimum allowing the specimens to cool from 1500°C to about 900°C in 7 - 8 minutes. All power was then switched off and the furnace allowed to cool.

#### B. Heat treatment under stress

The heat treatment cycles employed in these tests have been the same as in those without an applied stress.

The main problem in carrying out torsional tests on tungsten base materials is the hardness and brittleness of the material which makes machining operations (except for grinding) difficult. The attachment (by welding) of end pieces, suitable for gripping may be unsatisfactory due to the

severe loss of properties in the tungsten arising from grain growth near the weld. It was decided therefore to use a specimen that would be easily prepared by grinding and to fix it by a mechanical locking system in the specimen grips.

Since the specimen grips were to be subjected both to the heat-treatment temperatures and the load, the choice of suitable materials for them was limited either to a refractory metal or to a cooled system using copper or steel, or to graphite. Since no suitable refractory metal was available, and since, in any case, the machining problems would have been considerable, this possibility was discounted. The choice lay therefore between a cooled grip and a graphite one. On the grounds of simplicity the graphite was chosen initially and has proved satisfactory. The method of fixing, and the specimen dimensions, are shown in Fig. 2. In order to determine if the graphite could withstand the stress involved, an assembly was made, using a carbon steel specimen, and tested in a torque machine at room temperature. The graphite successfully withstood applied stresses of twice those anticipated under service conditions. It was considered therefore that, having regard to the improved mechanical properties of graphite at high temperatures over those at room temperature, the loading system would be satisfactory. This has proved true.

Since the applied torques - up to about 8 lbs. ins. - would be only about one-tenth of those used for the graphite tests on the torsional rig, it was necessary to increase the sensitivity of the load measuring device. Formerly, this measurement was made by measuring the deflection under load of three identical flexible beams equally spaced at  $120^\circ$  to each other. The sensitivity was increased by removing two of these beams and then measuring the deflection of the remaining one. The rig was recalibrated by simultaneously noting the deflection of the beam and the elastic strain produced in a steel specimen of accurately known dimensions.

The specimen was allowed to soak for 1 minute at temperature before the application of the load and to recover for 1 minute after removal of the load before the temperature was lowered.

## Results

### A. Heat treatment without stress

- (1) Short time heat-treatments (15 minutes) at  $1700^\circ\text{C}$  and  $1900^\circ\text{C}$ .

The treatments have been successfully carried out. Some blue/purple colouration of the specimen surface has been observed.

(ii) 11 hour treatment at 1900°C.

This treatment was stopped by a fuse failure after 10½ hours. As this occurred during the night the specimens were, therefore, cooled rapidly. In order to avoid any possible changes in microstructure due to this rapid cooling, the specimens were re-heated the next morning to 1900°C, allowed to soak for a further 15 minutes, and then cooled by the standard procedure.

(iii) Heat treatment at 2500°C for 15 minutes

Owing to incorrect pyrometer focusing the first set of specimens intended for heat-treatment at this temperature were inadvertently heated considerably higher. This temperature was believed to be approaching 2700°C and the specimens were at that temperature for some 20 - 25 minutes. Considerable fusion occurred between the specimens.

A second batch was successfully treated at the correct temperature when little adhesion between adjacent specimens occurred. At these high temperatures it is anticipated that some tungsten carbide may be formed and this point is being investigated by Aeon Laboratories.

B. Heat treatment under stress

To date two treatments have been carried out, one at 1700°C and another at 1900°C. The full load was reached in about 1 minute after the start of application. The cycle at temperature was therefore:-

- Reach temperature
- 1 minute soaking time
- 1 minute of rising load
- 14 minutes soaking under full load
- Removal of load
- 1 minute recovery time
- Temperature lowered 50°C

The torque applied in both these experiments has been 8 lbs. ins., maintained within  $\pm 2.5\%$ . For the gauge diameter of 0.090 ins. the intensity

of the surface stress,  $f_s$ , is calculated as follows:-

$$\text{Torque} = f_s \frac{J}{R}$$

$$\therefore f_s = \frac{RT}{J} = \frac{0.045 \times 8}{6.44 \times 10^{-6}} = 5.6 \times 10^4 \text{ p.s.i.} = 25 \text{ t.s.i.}$$

where  $J$  is the polar moment of inertia  $\frac{\pi R^4}{2}$ , and  $R$  the radius

(see A. Morley, Strength of Materials, 11th edition, p.323)

The permanent strain produced at  $1700^\circ\text{C}$  was  $0.034^\circ$  twist and at  $1900^\circ\text{C}$  about  $0.5^\circ$ . The development of the strain at  $1700^\circ\text{C}$  is given in Table 1.

### Conclusions

(a) The high temperature apparatus originally developed for testing graphite can be used successfully for the heat treatment of tungsten. The possibility of carbide formation must always, however, be considered.

(b) The loading assembly for heat-treatment under stress has been shown to be satisfactory.

TABLE 1

Change in strain with time in tungsten-thoria (%) at 1700°C under torque of 8 lbs. ins.

Twist on loading 0.341°

| Time of application<br>of full load (mins.) | Creep Twist<br>° | Creep Shear Strain<br>10 <sup>-5</sup> ins./ins. |
|---------------------------------------------|------------------|--------------------------------------------------|
| 0                                           | 0                | 0                                                |
| 1                                           | 0.039            | 3.4                                              |
| 2                                           | 0.056            | 4.9                                              |
| 3                                           | 0.057            | 5.0                                              |
| 4                                           | 0.065            | 5.7                                              |
| 5                                           | 0.065            | 5.7                                              |
| 10                                          | 0.117            | 10.2                                             |
| 15                                          | 0.122            | 10.6                                             |
| After 1 minute recovery<br>with no load     | 0.034            | 3.0                                              |

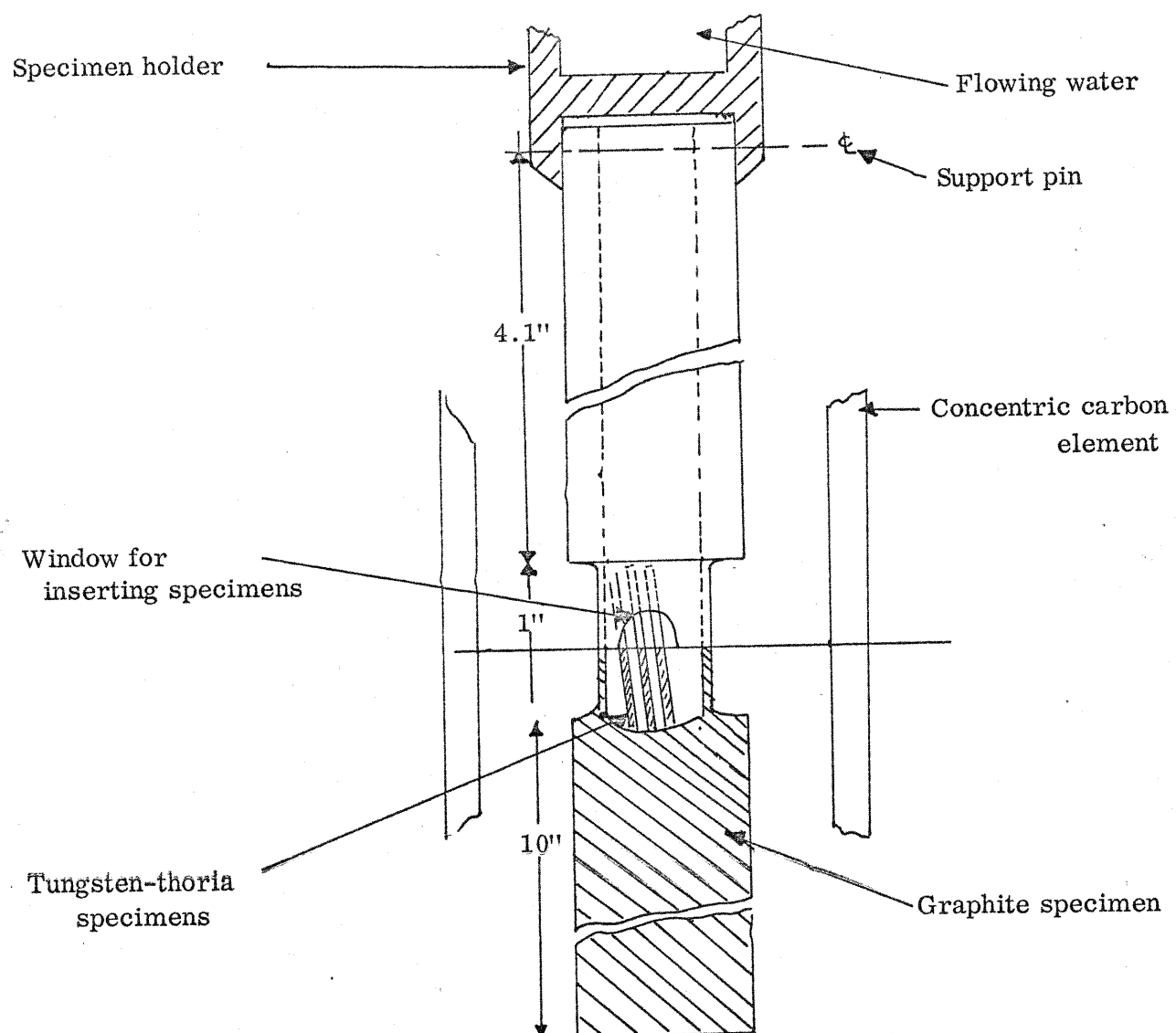


Figure 1. Part section view of arrangement for heat-treatment without applied stress

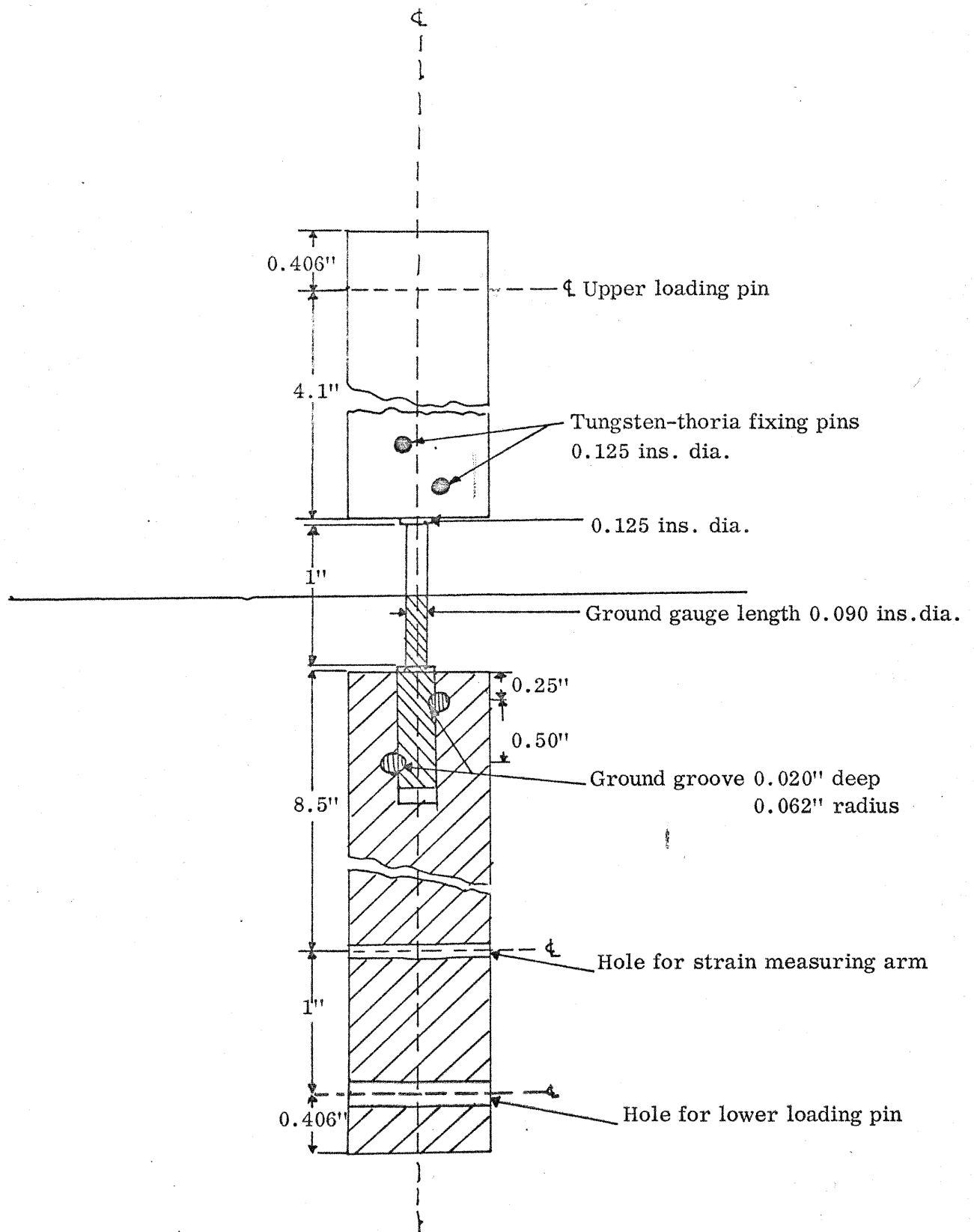


Figure 2. Assembly for heat-treatment under load.