Material Properties of Tantalum including High Cycle Fatigue

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There is a need for Tantalum (Ta) design data for ISIS spallation target design, especially for high cycle fatigue. The target operates under a high duty cycle of pulsed heat load. Very little such information exists in the literature for this material. HIPed (Hot Isostatic Pressed) Ta is used in the target manufacture and so data for As Received (A/R) and HIPed material is required. Following a comprehensive tensile test programme to establish batch properties, suitable fatigue test specimens have been carefully prepared to ASTM specifications. Great care has been taken in the selection of the fatigue test, load/strain control, frequency and monitoring in order to gain good quality and useful data. A small number of specimens have been cycled at various loads up to 480 million cycles before failure. Cold creep has been found to be an issue and investigated. The preparation, test methods and results for the first two A/R specimens are presented.

KEYWORDS: Tantalum, Ta, high cycle fatigue, HIP, material properties

1. Introduction

Tantalum is often chosen for a spallation target material, either solid or as a thin cladding especially if water cooled. ISIS, Los Alamos and the proposed SNS 2nd Target are examples of this. Although its neutronic performance is slightly lower than Tungsten, it thereby prevents Tungsten/water/beam corrosion issues. In order to facilitate mechanical design of such a target, accurate property data is needed. In particular, there is very little fatigue data available. Tantalum is metallurgically a complicated refractory metal which is known to exhibit cyclic stress hardening and softening, further complicating the measurement of fatigue properties. It is highly desirable to get applicable fatigue data for the particular ISIS target operating regime. The main source of relevant data is that of Papakyriacou et al 1) which surprisingly indicates a very high fatigue life (~100 million cycles) at room temperature above UTS load at 100 Hz although with rotating bend type fatigue testing which is less representative of ISIS loading and is known to give a lot of scatter.

2. ISIS Target configuration and operating regime

Bulk tungsten plates (Target Station 1) and rod (TS2) are used with a thin Layer of Ta (0.8 – 2mm) depending upon the design configuration. This assembly is HIPed together at high temperature which locks in extremely high residual tension stresses in the Ta due to differential Ta/W contraction. The proton pulse frequency (and therefore heat input frequency) is 50Hz (although every 5th pulse is generally diverted to TS2) which induces 160 KW of pulsed heating in TS1. The lifespan for a TS1 target is around 2500 mA hours, which depending on the maintenance periods, equates to a 5 year typical lifetime spanning 2700 million cycles. The simulated periodic Von Mises stress is about +195 MPa mean
value and amplitude $\sim5$ MPa. The predicted Ta operating temperature spans a wide range depending on target configuration up to a maximum of 172 degrees C.

3 Material selection

This programme intentionally uses typical commercially available pure Ta such as is used for target manufacture, but with full QA and inspection including chemical composition. Tensile and hardness tests have been carried out to establish basic mechanical properties as a baseline for the fatigue tests. The target manufacture uses tube, rod and plate forms of Ta which will affect the grain size and properties but to get initial results, this study uses 13mm diameter annealed rod stock. These have been produced by powder metallurgy and sintering.

4 Tensile results

| Sample Id | Modulus (MPa) | Upper Yield (MPa) | 0.2% PS (MPa) | 0.5% Strain (MPa) | UTS (MPa) | Elong. (%) |
|-----------|---------------|------------------|---------------|------------------|-----------|-----------|
| 1         | 182           | 278              | 212           | 207              | 295       | 50.5 *    |
| 2         | 173           | 274              | 213           | 206              | 296       | 63.8      |
| 3         | 172           | 200              | 170           | 168              | 264       | 71.7      |
| 4         | 177           | 266              | 198           | 190              | 284       | 66.2 *    |
| 5         | 182           | 262              | 201           | 192              | 285       | 69.0 *    |

*indicates the specimen failed outside the middle 50%, but still within the gauge length. (There is no reason why an un-notched specimen should fail exactly in the middle if prepared uniformly)

Baseline data:

- Yield (0.2%) $\sim200$ MPa ($170 – 213$)
- Average UTS $284$ Mpa
- Lower bound UTS $264$

5 Fatigue Specimen Preparation

As received (A/R) and HIPed specimens will be tested in the complete programme, these first two specimens are A/R. They are machined to ASTM E466-15 dimensions for a circular cross section specimen with continuous radius (see Fig.1) and the gauge length is longitudinally polished and measured to have good longitudinal RA $= 0.2469 \mu$m. This was the limit achievable before smearing occurred with this ductile material.

6 Test Specification

This initial study is carried out at room temperature and for simplicity the specimen temperature was not monitored. Unlike an ultrasonic test, internal heating at the maximum frequency $\sim130$ Hz is not expected to increase specimen temperature significantly. Monitoring during later testing indicated a temperature of no more than 1C above test house ambient. Furthermore, at this homologous temperature of about $0.09 T_m$
the fatigue properties of this refractory metal are not expected to vary significantly with temperature changes of a few degrees.

A trial specimen and then 2 main specimens were tested at a range of loads in high cycle fatigue. The default test duration is 100 million cycles but it was lengthened to get data on longer duration when opportunity existed. A (10 MPa – Max Load) sinusoidal Tension-Tension load was used to correspond to the predicted target load regime which is always positive due to the high residual HIP stress (see above). Although the ISIS duty cycle is 50 Hz it was important to run these tests as fast as possible to get timely results at feasible cost, and the resonant machine frequency of around 100 Hz is comparable unlike ultrasonic testing. The test was based on the ASTM E466 Force controlled fatigue test specification with modifications. The initial plan was to start on a servo-hydraulic machine at ~0.5 Hz with extensometers, and then move to a resonant test machine to run at ~100 Hz once the plastic strain had decreased. A combination adaptive strain/load control methodology was initially used.

6.1 Ramp/Pre-conditioning
There was concern that for tests at high load the specimen might fail prematurely during the first few cycles, and so preconditioning was applied for the 2nd test where the load is above expected yield. 20,000 cycles were run at each of ~50, 60, 70, and then 80% of the max load.

7. Results
A trial Ta specimen of same dimensions, material condition and finish was first used to test the experimental set-up and procedure. This proved invaluable to check the test methodology.

7.1 Early Fatigue Results using trial specimen
Figure 2 shows the load/displacement behavior which is as expected, and Fig. 3 shows in close up the load repeatedly dropping off as it strains plastically before the adaptive control increases the target displacement. However, at 6000 cycles it can be seen (Fig. 4) that the extension was just increasing indefinitely under the fatigue load so the test was stopped, and a fixed load was applied to the same specimen thereafter. Similar further extension behaviour was observed (Fig.5).

The extended time scale of this deformation is too long to just be yielding. Unexpectedly, cold creep is occurring, and this was investigated by testing another simple tensile Ta sample see Figs 6,7,8. After a slight anomaly at 50 MPa, increasing levels of creep are shown at higher loads from 150 MPA. The yield range is 170 – 213 MPa so we are seeing creep from just below yield. The Sandia report 2) discovered subsequently broadly backs up this finding of increasing creep in Ta at high stress levels and room temperature from about yield load.
FIG. 2. Trial Ta Specimen : Load and Extension vs Cycles

FIG. 3. Trial Ta Specimen : Close-up Cycles 700 - 800
FIG. 4. 10 -264 MPa Fatigue Load

FIG. 5. Continuation as a Fixed Load 264 MPa

FIG. 6. Creep specimen

FIG. 7. Creep Test Load Profile

FIG. 8. Creep Test Stress/Strain
7.2 Specimen 1

In order to limit the effects of unwanted creep, a fatigue test was carried out purely on a resonant ‘Vibrophore’ machine at 130 Hz, initially at (10 - 100 MPa) for 160 million cycles, and then continued at increasing max load as shown in table II until failure at 280 MPa after 480 million cycles with a crack. The fracture face Fig. 13 is clearly fatigue failure. The machine had no displacement output, hence grip separation was used as a crude indication. It is not noted from the very beginning but can be assumed to be zero. This separation and the resonant frequency show an effect starting from about 200 MPa and 400 million cycles.

Table II: Specimen 1 Results

| MPa  | Grip separation (mm) | Resonant Frequency (Hz) | Cycles   | Total        |
|------|----------------------|-------------------------|----------|--------------|
|      | Start                | Finish                  | ∆        | Start        | Finish       |          |          |
| 100  | -                    | -                       | -        | 129.8        | 130          | 150,248,416 | 150,248,416 |
| 120  | -                    | -                       | -        | 129.9        | 129.7        | 79,363,769  | 239,214,185 |
| 135  | 16.7                 | 16.7                    | 0        | 129.8        | 129.8        | 85,734,490  | 325,948,675 |
| 150  | 16.7                 | 16.7                    | 0        | 129.8        | 129.7        | 50 million  | 375,948,675 |
| 160  | 16.7                 | 16.7                    | 0        | 129.8        | 129.7        | 10 million  | 375,948,675 |
| 170  | 16.7                 | 16.7                    | 0        | 129.8        | 129.8        | 10 million  | 375,948,675 |
| 180  | 16.7                 | 16.71                   | 0.01     | 129.8        | 129.8        | 10 million  | 375,948,675 |
| 190  | 16.71                | 16.72                   | 0.01     | 129.7        | 129.7        | 10 million  | 375,948,675 |
| 200  | 16.72                | 16.75                   | 0.03     | 129.8        | 129.8        | 10 million  | 375,948,675 |
| 210  | 16.75                | 16.85                   | 0.08     | 129.8        | 129.8        | 10 million  | 375,948,675 |
| 220  | 16.8                 | 16.9                    | 0.07     | 129.2        | 129.0        | 10 million  | 375,948,675 |
| 230  | 16.9                 | 16.95                   | 0.05     | 129.0        | 129.8        | 10 million  | 375,948,675 |
| 240  | 16.95                | 17.05                   | 0.10     | 128.8        | 129.0        | 10 million  | 375,948,675 |
| 250  | 17.05                | 17.15                   | 0.10     | 128.6        | 128.4        | 10 million  | 375,948,675 |
| 260  | 17.15                | 17.4                    | 0.25     | 128.4        | 127.8        | 10 million  | 375,948,675 |
| 270  | 17.4                 | 17.6                    | 0.2      | 127.8        | 127.4        | 10 million  | 375,948,675 |
| 280  | 17.6                 | -                       | -        | 127.4        | 125          | 1,401,213   | 487,349,888 |

Table III: Specimen 1 Dimensions

| Specimen 1 | Dia | Overall Length |
|------------|-----|----------------|
| Before test| 4.9 | 74.07          |
| After Test | 4.5 | 75.31          |
| Change     | 0.3 | 1.24           |
7.3 Specimen 2
This was run at 252 MPa stress for 260 million cycles (see table IV) without failure although there was a total extension of 0.56 mm. There was a very gradual drift down of resonant frequency by 1Hz until about 20 million cycles which will be due to the plastic extension. At this point some instability in the control loop started and gave variations of +/- 4MPa (1.5%) in the stress limits but did not affect the mean stress. This small variation is not expected to affect the test results, although extra weight was added on the advice of specialist technical support to eradicate it with a consequent change in frequency as noted. Some foam damping was also added to lower the Q but removed because ineffective. LVDTs were attached for the early part of the test but the data logger and electronics were insufficiently fast to capture data during fatigue.

Table IV: Specimen 2 Results

| Cycles   | Frequency (Hz) | Notes               | Cycles   | Frequency (Hz) | Notes               | Cycles   | Frequency (Hz) | Notes               |
|----------|----------------|---------------------|----------|----------------|---------------------|----------|----------------|---------------------|
| 4,000    | 130.9          |                     | 170,000  | 130.2          | LVDT’s removed      | 24,273,567 | 129.6          |
| 10,000   | 130.9          |                     | 220,000  | 130.1          |                     | 26,421,567 | 129.4          |
| 16,000   | 130.8          |                     | 290,000  | 130.0          |                     | 56,883,567 | 129.4          |
| 30,000   | 130.6          |                     | 460,000  | 130.0          |                     | 56,888,567 | 103.9 Extra weight added to resonant circuit |
| 40,000   | 130.6          |                     | 760,000  | 129.9          |                     | 73,631,567 | 103.9          |
| 47,000   | 130.5          |                     | 1,100,000| 129.8          |                     | 96,114,567 | 103.9          |
| 70,000   | 130.4          |                     | 3,200,000| 129.8          |                     | 117,519,567| 104.5 Damping added to resonant circuit |
| 90,000   | 130.3          |                     | 4,340,000| 129.7          |                     | 190,983,567| 104.6          |
| 110,000  | 130.2          |                     | 20,200,000| 129.8 Peak stress +/- |                     | 259,783,567| 104.8          |

Table V: Specimen 2 Dimensions

| Specimen 2 | Diameter mm | Overall Length mm |
|------------|-------------|-------------------|
| Before test| 4.957       | 74.14             |
| After Test | 4.804       | 74.70             |
| Change     | -0.153      | 0.56              |

FIG. 13. Specimen 2 Test Set-up
8. Conclusions

The 1st specimen with an increasing load applied survived a total of 487 million cycles to fail at 280 MPa. The intention was to initially show a clear endurance at 100 MPa and this was achieved with 150 million cycles. A high fatigue life was subsequently demonstrated at each of 120, 135, and 150 MPa before doing 10 million cycles at each increasing 10 MPa interval to search for a failure stress (see Table II). The ultimate failure at 280 MPa may be due to the load level but another factor will clearly be the overall high number of cycles and underlying fatigue damage. This final result is only indicative because as stated the load was increased in stages during the test.

The 2nd specimen ran for 260 million cycles at constant max fatigue load of 252 MPa with no sign of failure and a final extension of just 0.56 mm.

Although only a small number of specimens, these early results start to indicate that Ta can survive very high numbers of cycles (well above 100 million) at high loads around the UTS in this loading regime.

Surprisingly, cold creep has been experienced and confirmed by investigation. It is not known to be significant in the ISIS target Ta, which runs at 50 Hz, and so the testing was adapted to purely higher frequency loading to mimic the ISIS regime, and minimize the influence of creep in order to focus on the effect of fatigue damage. This was successful and while creep was dominant in the first low frequency (~1Hz) tests done, there was clearly far less at the higher frequency tests above 100 Hz as shown by the relatively low overall plastic extension despite extremely high number of cycles. The conclusion is that creep needs an initiation time, since the total time at high load is just as high with higher frequency loading.

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References

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