**SPALLATION THRESHOLD TESTS
ON NIOBIUM, TANTALUM,
AND ZIRCONIUM**

G. P. HANNEMAN

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By

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ABSTRACT

Niobium, tantalum, and zirconium specimens were heated to elevated temperatures in less than 8.0×10^{-6} sec by capacitor discharge. The ratio of heating time to thermal expansion time was maintained at unity or less to induce large tensile stresses. Single or multiple spallation was determined to occur in a plane normal to the long axis when temperature rose above a certain threshold. Spall threshold was found to be in the range of 1730 to 1860 °C for niobium, 1900 to 2140 °C for tantalum, and 1150 to 1200 °C for zirconium.

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INTRODUCTION

A spallation threshold study was performed on three refractory metals - niobium, tantalum, and zirconium. The approximate temperature above which each of these metals would fracture by spallation was determined.

When a narrow metallic strip or wire is uniformly heated so rapidly that the heating to expansion time ratio is equal to one or less, a significant compressive stress develops. The resulting rapid lengthwise expansion produces a tensile stress throughout the strip. If this stress is relatively low, the strip will simply cycle between compression and tension until the oscillation stops. At sufficiently high peak temperatures and tensile stresses, however, the strip will fracture or spall in a plane roughly perpendicular to its long axis.

PROCEDURE

MATERIAL

Two nominally pure niobium strips were obtained from cold-rolled 0.020 in. thick sheet by shearing and hand grinding to the final 0.020 x 0.100 x 2.00 in. dimensions. Two nominally pure zirconium strips were similarly prepared from 0.030 in. thick sheet to 0.030 x 0.100 x 2.00 in. dimensions. Six tantalum specimens were cut from annealed, 99.8% pure, 0.040 in. diam wire and ground to 2.00 in. length.

EQUIPMENT

A diagram of the test equipment is illustrated in Figure 1. Energy for spallation was supplied from a bank of three high energy capacitors capable of storing up to 5600 J at 20,000 volts. Short, low-inductance buses led to the specimen mount in a nitrogen atmosphere, gloved box. Here the refractory metal specimens, mounted between the terminal ends of the buses, were contacted only through 0.001 in. diam copper wires at each end. A thin exploding wire switch, fitted into a thin slot all the way through one bus, was the mechanism used to start each test. During the first part of the capacitor discharge, of micro-sec duration, relatively low resistance contacting arcs were formed at the specimen ends by vaporization and ionization of the copper wire. Thus the specimen ends were free to thermally expand so that the spallation phenomenon could occur. This procedure permitted greater than 95% energy deposition in less than 8×10^{-6} seconds.

MEASUREMENTS AND ANALYTICAL METHODS

Two test events were recorded. One was the differential output of a chromel/alumel thermocouple intrinsically butt-welded to the specimen. The other was the electronically-integrated output of a small inductive pickup coil located near one of the buses. The oscilloscope recording of a typical test shows representative current and thermocouple traces (Figure 2). The recording yields the total electrical resistance and inductance of the discharge circuit and, thus, the energy deposit with time. In addition, the peak temperature reached is also indicated.

FIGURE 1. Diagram of Capacitor Discharge Test Equipment Used in Spallation Threshold Studies

FIGURE 2. *Typical Oscilloscope Recording of the Dual-Beam Traces of Integrated Current and Thermocouple Output. Upper Trace: 0.5 V/cm at 5×10^{-6} sec/cm. Lower Trace: 10 mV/cm at 0.1 sec/cm.*

The technique for determining the peak test temperature, attained when the thermocouple failed, involved establishing a graphical relationship based on tests in which temperature measurement was obtained. The analytic form of the relationship is:

$$\frac{\text{Total Stored Energy}}{\text{Specimen Mass}} \times \frac{\text{Avg Specimen Resistance}}{\text{Avg Total Circuit Resistance}} = \text{Graphical Function of Temperature}$$

This relationship resulted in an ordinary total heat input versus temperature curve. The Average Specimen Resistance term was evaluated at several points during heating because of variations, depending on the material and on the final temperature reached, of up to 5 times its starting value.

SPECIMEN SIZE REQUIREMENTS

The surface heating effect was essentially eliminated in these tests by keeping the ratio, R_{HF}/R_{DC} , fairly close to 1.00. Here, R_{HF} is the higher resistance of the specimen due to reduced current near the conductor center, a phenomenon of

high frequency alternating current, while R_{DC} is the direct current resistance. By using a standard graph relating this ratio to a function of current frequency, electrical resistivity, and conductor cross-section dimensions, a specimen cross section could be selected which would hold the ratio between 1.02 to 1.04 for all three metals for tests over 1000 °C, where resistivities were relatively high.

A 2 in. specimen length was chosen for the three test materials to provide expansion time sufficient to achieve a 97% energy deposit for niobium, 99.5% for tantalum, and 99% for zirconium before any tensile stress could develop.

RESULTS AND DISCUSSION

The testing of niobium, tantalum, and zirconium over a wide range of temperatures below the spall threshold produced measurable increases in cross section (Tables I, II, and III). This compressive plastic deformation indicated that the elastic limit was exceeded during the initial compressive loading. Plastic extension may have occurred during the tensile loading immediately following. However, measurements to determine the amount of this extension were not made. Sufficiently high stresses in the tensile cycle would result in spallation, frequently accompanied by necking.

As shown in Table I, Niobium Specimen No. 1 was tested seven times and No. 2 five times at successively higher temperatures until spallation occurred or testing was stopped (Figure 3). Although significant specimen thickening was found for tests at 1080 °C and above, test temperatures through 1730 °C did not cause spallation. The lowest temperature at which spall occurred was 1860 °C. Thus the spall threshold for niobium lay within the range 1730 to 1860 °C.

TABLE I. *Physical Change in Niobium Strips as a Function of Sudden Temperature Rise from Room Temperature*

<u>Test No.</u>	<u>Peak Temp, °C</u>	<u>Initial Condition</u>	<u>Cross-Section Change from Prior Test, %</u>	<u>Final Condition</u>
<u>Specimen No. 1</u>				
N1-1	420	Cold-worked	+0.2	No change observed
N1-2	670	Cold-worked	-0.5	No change observed
N1-3	930	Cold-worked	+0.2	No change observed
N1-4	1080	Annealed*	+2.2	Thickened along centerline while strip narrowed
N1-5	1370	Annealed*	+0.1	Thickened along centerline while strip narrowed
N1-6	1720	Annealed*	+4.2	Thickened along centerline while strip narrowed
N1-7	1860	Annealed*	+6.1	Thickened along centerline while strip narrowed plus single break near one end
			(+12.5 cumulative)	
<u>Specimen No. 2</u>				
N5-1	190	Cold-worked	ND**	No change observed
N5-2	400	Cold-worked	ND**	No change observed
N5-3	740	Cold-worked	ND**	No change observed
N5-4	1190	Cold-worked	ND**	No change observed
N5-5	1730	Annealed*	(+12.5 cumulative)	Thickened along centerline while strip narrowed

* Annealed by previous test
 ** Denotes no data taken

Separate tantalum specimens were used for each of the T1-1 to T1-5 tests (Table II and Figure 4), while one specimen was used in the T5-1 to T5-4 series. The highest test temperature reached without spall was 2140 °C, while the lowest with spall was 1900 °C. Therefore, the tantalum spall threshold was placed between these two temperatures.

TABLE II. *Physical Changes in Tantalum Wires as a Function of Sudden Temperature Rise from Room Temperature*

Test No.	Peak Temp, °C	Initial Condition	Cross-Section Change from Prior Test, %	Final Condition
T1-2	1890	Annealed	+2.9	No change observed
T1-4	2140	Annealed	ND*	Bent into curve
T1-5	2200	Annealed	ND*	Five breaks
T1-3	2260	Annealed	ND*	About nine breaks
T1-1	3000	Annealed	ND*	Fine powder from complete melting and scattering
T5-1	390	Annealed	ND*	No change observed
T5-2	840	Annealed	ND*	No change observed
T5-3	1380	Annealed	ND*	No change observed
T5-4	1900	Annealed	ND*	Single break near one end

* Denotes no data taken

As shown in Table III and Figure 5, Zirconium Specimen No. 3 was tested eight times and No. 4 six times at successively higher temperatures until fracture occurred. The specimens remained intact up to 1150 °C, while fracture occurred at ~1200 °C, indicating the zirconium spall threshold to be between 1150 to 1200 °C.

TABLE III. *Physical Changes in Zirconium Strips as a Function of Sudden Temperature Rise from Room Temperature*

<u>Test No.</u>	<u>Peak Temp, °C</u>	<u>Initial Condition</u>	<u>Cross-Section Change from Prior Test, %</u>	<u>Final Condition</u>
<u>Specimen No. 3</u>				
Z1-1	590	Cold-worked	+0.8	Still intact
Z1-3	740	Cold-worked	-0-	Still intact
Z1-4	860	Annealed*	+0.1	Still intact
Z1-5	920	Annealed*	-0.4	Still intact
Z1-6	980	Annealed*	+0.1	Still intact
Z1-7	1150	Annealed*	ND**	Still intact
Z1-8	~1200	Annealed*	ND**	Broken off ~ 0.1 in. from end plus other end (~ 0.1 in.) completely melted
<u>Specimen No. 4</u>				
Z5-1	220	Cold-worked	ND**	Still intact
Z5-2	230	Cold-worked	ND**	Still intact
Z5-3	475	Cold-worked	ND**	Still intact
Z5-4	750	Cold-worked	ND**	Still intact
Z5-5	1140	Annealed*	ND**	Still intact
Z5-6	1300	Annealed*	ND**	Two ragged breaks-- one very near center, other ~ 0.1 in. from end

* Annealed by previous test

** Denotes no data taken

FIGURE 3. *Niobium Strips Tested for Spallation Threshold,*
~1X

FIGURE 4. *Tantalum Wires Tested for Spallation Threshold,*
~1X

FIGURE 5. *Zirconium Strips Tested for Spallation Threshold,*
~1X

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