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Thermal Shock Experiment (TSEX):
A "Proof-of-Principle" Evaluation of the
Use of Electron Beam Heating to Simulate the
Thermal Mechanical Environment Anticipated for the
First Wall of the Reference Theta-Pinch Reactor (RTPR)

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THERMAL SHOCK EXPERIMENT (TSEX): A "PROOF-OF-PRINCIPLE"
EVALUATION OF THE USE OF ELECTRON BEAM HEATING TO SIMULATE THE
THERMAL MECHANICAL ENVIRONMENT ANTICIPATED FOR THE FIRST WALL
OF THE REFERENCE THETA-PINCH REACTOR (RTPR)

by

P. E. Armstrong and R. A. Krakowski

ABSTRACT

The results of a "proof-of-principle" Thermal Shock Experiment (TSEX), designed to simulate the thermal mechanical response of insulator-metal composite first walls anticipated for pulsed high-density fusion reactors, are given. A programmable 10-kV, 1.0-A electron beam was used to pulse repeatedly (0.30-mm)Al₂O₃/(1.0-mm) Nb-1Zr composite samples 200-300 K, relative to a base-line temperature of 1000 K. The experimental goals of TSEX were established relative to the first-wall environment anticipated for the Reference Theta-Pinch Reactor (RTPR). A detailed description of the TSEX "proof-of-principle" apparatus, experimental procedure, and diagnostics is given. The results of extensive thermal analyses are given, which are used to estimate the thermal stresses generated. Although little or no control was exercised over the sample fabrication and thermal history, one sample experienced in excess of 800 thermal cycles of ~ 250 K at ~ 1000 K, and the results of optical and SEM examination of this specimen are presented. The resistance of this sample to macroscopic failure was truly impressive. Recommendations for the construction of an apparatus dedicated to extensive testing of first-wall composites are given on the basis of these "proof-of-principle" TSEX results.

1. INTRODUCTION

Fusion reactor concepts based upon pulsed, high-beta confinement schemes, such as that embodied in the Reference Theta-Pinch Reactor (RTPR) design,^{1,2} will subject the first wall to significant thermal excursions. Although a cursory treatment of the related thermal stress and cyclic fatigue problem might show that these problems are insurmountable,³ more detailed analyses^{4,5} clearly point the way to realistic solutions to the thermal mechanical first-wall problem.

For reasons briefly summarized in Sec. 2., the first wall envisaged for the RTPR must be coated with a thin layer of dielectric material, and a (0.3-mm) Al_2O_3 /(1.0-mm) Nb-1Zr composite is being considered as a reference system. Thermal mechanical testing of first-wall materials under actual conditions envisaged for the fusion reactor is not possible at present. For this reason conceptual reactor designs have relied almost entirely upon sophisticated thermal stress computations.⁵ A purely analytic approach to the RTPR first-wall response, no matter how detailed, is limited; experimental and developmental efforts on a modest level are needed to substantiate or to disprove the predictions made by these thermal mechanical analyses. For this reason a pulsed heating apparatus was constructed to examine the applicability of electron beam (EB) heating to simulating the first-wall thermal mechanical response. Reported herein are the results of "proof-of-principle" tests of this EB technique as applied to the (0.3-mm) Al_2O_3 /(1.0-mm) Nb-1Zr first-wall system. Since the major goal of this experiment was the development of the EB simulation technique for the simulation of the first-wall thermal mechanical response, little or no control of test specimen fabrication and exposure history was made. Preliminary results on a single sample are reported, however, and recommendations are made for a more definitive Thermal Shock Experiment (TSEX).

2. SUMMARY DESCRIPTION OF RTPR PULSED OPERATION

A major goal in the development of the TSEX apparatus was the simulation of first-wall thermal mechanical environment anticipated for pulsed high-beta fusion reactors. For this reason a brief description of the most completely documented conceptual design for a reactor of this kind is given.

An essential element in the operation of the RTPR¹ is plasma preheating by means of a fast-rising magnetic field (~ 1.0 T in ~ 1.0 μ s). These fields

would be generated by a low-inductance, fractional-turn coil located outside a high-temperature blanket system. The preheated plasma (~ 1 keV) is subsequently compressed adiabatically by a slow-rising magnetic field (~ 7 - 10 T rising in ~ 30 ms). The multiturn, adiabatic compression coil is located radially outward from the implosion heating coil. The physical relationship between the plasma chamber, blanket structure, and magnetic coils is depicted on Fig. 1 for a ~ 2 -m-long RTPR module. Approximately 200 such modules would form the RTPR torus.

For the magnetic flux generated by the implosion heating coil to penetrate the blanket and to implode the plasma on a ~ 1.0 - μ s time scale, the blanket module must not form an electrically conducting path in the azimuthal or "theta" direction. Consequently, the RTPR blanket would be comprised of a number of azimuthal segments, which must be electrically insulated from each other. The blanket structure, therefore, serves as a concentrator of magnetic flux on the time scale of the plasma implosion (~ 1.0 μ s); the blanket structure in no way is "hard wired" into the implosion heating circuit but only is coupled inductively. The azimuthal electric field that "drives" the implosion heating coil, consequently, is induced across each of the azimuthal blanket segments. Hence, the electrical insulation attached to each blanket segment must be of high pulsed-dielectric quality (~ 100 kV/cm). This electrical insulation must partially extend into a first-wall region, as is shown in Figs. 1-2. It is this region which has been the focus of appreciable thermal mechanical analysis.⁵

During the thermonuclear burn the RTPR first wall will be bombarded with bremsstrahlung, neutron, and gamma radiation. Figure 3 gives the time dependence of neutron (20-MeV/n) energy flux, bremsstrahlung temperature, and bremsstrahlung energy flux for a reference thermonuclear burn. During the ~ 400 -ms burn, particle diffusion and charge-exchange rates are calculated to be small. After the burn the plasma internal energy (~ 6 MJ/m) must be conducted to the first wall by means of a neutral gas layer⁶ on a 2-4-s time scale. This "plasma-dump" phase of the RTPR burn cycle may also result in a significant heat flux.⁵

A generalized scenario for the RTPR first-wall design task is depicted in Fig. 4. The major approaches being considered to satisfy the constraints summarized in Fig. 4 are: (1) reconfiguration of the first wall, (2) careful selection and re-engineering of candidate first-wall materials and (3)

tailoring of the RTPR operating point within realistic plasma physics constraints to conform to first-wall engineering limitations. The results presented in Ref. 5 pertain only to item (1). Item (2) is not amenable to a calculational approach, although a research development effort at LASL addresses the question of first-wall materials selection and re-engineering.⁷ A detailed reassessment of the RTPR operating point, insofar as the required implosion-heating voltages and first-wall energy densities are concerned, has been conducted in a more generalized context.²

Within the "configuration" scenario (Fig. 4) the approach adopted for the RTPR is twofold. First, a number of new first-wall configurations have been proposed and are classified as: (1) bonded or unbonded laminated composites (laminates), (2) all-ceramic structures, and (3) protective and/or sacrificial surfaces interposed between the first-wall and the plasma ("bumpers" or "fenders"). Figure 2 illustrates a number of first-wall configurations being

Fig. 1.

Isometric view of the geometric relationship between RTPR blanket structure, plasma chamber, and magnetic coils. Note that the segmented blanket is in no way "hard wired" to the implosion-heating circuit.

Fig. 2.

Schematic diagram of first-wall configurations being considered for the RTPR.

considered. Secondly, a more quantitative failure criterion for the first wall is identified which is based upon the developing theories of brittle fracture of ceramic materials.^{5,8} Conventional thermoelastic stress analyses as an absolute design criterion appear to be inadequate; a more detailed understanding of geometry effects, the mechanics of brittle fracture, and an explicit criterion for the magnitude of fracture and debonding needed to make the RTPR inoperable is being developed.

Within this context, therefore, a means was investigated by which the first-wall thermal cycle could be simulated for a pulsed fusion reactor.

Fig. 3

Time dependence of neutron current and energy (ϕ_n and W_N), bremsstrahlung power (P_{BR}), plasma temperature (T_i) and plasma energy (W_{INT}) anticipated for the RTPR first wall. The compression magnetic field is B_0 and the stabilizing feedback power is P_{FB}^0 (Ref. 2).

Fig. 4.
Summary of LASL approach to the RTPR first-wall design.

Figure 5 depicts the thermal and stress transient calculated for the RTPR burn cycle given in Ref. 2. The TSEX task, therefore, requires a complex and cyclic thermal load to be programmed at the insulator side of a (0.3-mm) Al_2O_3 /(1.0-mm) Nb-1Zr composite which is maintained at a base temperature of ~ 1000 K. In addition the Nb-1Zr side of the composite test specimen must be cooled in a controlled and programmable fashion. Lastly, the thermal pulse history must be reproducible at a rate that permits the accumulation of many cycles over a reasonable time period.

3. EXPERIMENTAL RATIONALE

The heat fluxes associated with the RTPR operating point will be composed of bremsstrahlung radiation ($45.9 \text{ J/cm}^2/\text{pulse}$ in ~ 0.4 s) and plasma internal energy ($189.8 \text{ J/cm}^2/\text{pulse}$ in ~ 2 s); neutron/gamma heating represents a volumetric heat source amounting to $215.6 \text{ J/cm}^3/\text{pulse}$ and $203.6 \text{ J/cm}^3/\text{pulse}$ in the (0.3-mm) Al_2O_3 and (1.0-mm) Nb-1Zr, respectively. Although a

2000 J/cm²/pulse of fusion neutron energy impinges upon the first wall, only 1.3% of this energy will be absorbed by the first-wall composite; the absorbed neutron/gamma energy amounts to 26.9 J/cm²/pulse. Since the bremsstrahlung and plasma internal energy are the major causes of first-wall temperature rise and thermal stress, the first-wall thermal mechanical response can be simulated experimentally by a programmable surface heat flux.

To minimize configurational effects and to assure simplicity and reproducibility in sample preparation, a disk configuration was chosen for the TSEX test specimen. The major goal of this "proof-of-principle" test was to identify a reliable, pulsed source of energy to be applied to an Al₂O₃/Nb-1Zr sample in such a way as to approximate the complex time dependence of the RTPR first-wall energy fluxes. The thermal cycle occurs relative to a ~1000 K base-line temperature, and five pulsed energy sources were considered for TSEX:

- a. electron beam (EB) bombardment,
- b. laser heating (cw-CO₂, ruby),
- c. plasma heating
 - Hall gun
 - axial discharge,
- d. arc-image furnace, and
- e. resistance heating.

Although laser heating appears to be an ideal heating technique, the ~100-W/cm² (on sample) requirement implies a large and expensive system. Both plasma heating methods are simple and inexpensive, but the relatively high-density discharges (~1 torr) required to achieve the desired energy densities are not compatible with vacuum constraints. Arc-image heating may be as (or more) favorable than laser heating, but represents a high cost and complexity. Resistance heating of the metallic substrate does not inject the heat flux at the insulator-vacuum interface unless a susceptor is used. A sizable (10-kW) electron beam (EB) power supply was available and is capable of programmable energy deposition with a ~1-ms risetime. Unless a thin conducting coating is applied to the ceramic-vacuum interface, however, a metallic susceptor must also be used. For reasons of cost and expedition, therefore, the EB bombardment heat source was selected for the initial "proof-of-principle" phase of TSEX.

The heat transfer code AYER⁹ was used to examine the temperature transient induced in a (0.3-mm)Al₂O₃/(1.0-mm)Nb-1Zr composite when heated either

indirectly by an EB-heated susceptor (tungsten) or directly by an EB impinging upon a 5- μ m tungsten coating deposited on the Al_2O_3 insulator. The results of this analysis showed that the susceptor must be pulsed to near the melting point if the thermal risetime depicted in Fig. 5 was to be achieved. On the basis of these preliminary calculations, the susceptor concept was rejected, and direct EB heating of the specimen was selected for the TSEX "proof-of-principle" tests. Also, for these preliminary tests the Nb-1Zr side of the specimen was for all intents and purposes radiation cooled to avoid initially the problems of complex cooling systems during the TSEX "proof-of-principle" phase. A controlled cooling of the Nb-1Zr surface will be eventually required in TSEX if the full RTPR burn cycle is to be accurately modeled (Fig. 5).

4. EXPERIMENTAL EQUIPMENT

The experimental apparatus uses a swept electron beam to deposit energy onto the surface of 25-mm-diameter by 1-mm-thick Al_2O_3 /Nb-1Zr sample. Although the sample rests in vacuo on a water-cooled heat sink, the poor contact between sample and sink under vacuum conditions results essentially in radiation cooling as the dominant cooling mechanism. Adjustment of both the incident EB energy and the thermal resistance between source and sink resulted in thermal excursions at the vacuum- Al_2O_3 interface which approximated in risetime and magnitude that expected for a reactor first wall (Fig. 5); no attempt was made in the "proof-of-principle" experiments to simulate the actual temperature waveform, although the capability exists to program any time-dependent EB waveform with an intrinsic ~ 1 -ms time constant. The apparatus was previously used for EB zone melting of refractory metal crystals, and only the EB gun and sample heat-sink structure were added for this experiment. The plasma-arc-sprayed (0.3-mm) Al_2O_3 /(1.0-mm)Nb-1Zr samples were used as received from the materials technology group (CMB-6). The EB energy (voltage and current) and Al_2O_3 -vacuum interface temperatures were measured. Optical metallography was performed on some samples which received as many as 800 200-300 K thermal pulses at a ~ 1000 K base-line temperature.

4.1 Sample Chamber

Samples were thermally pulsed in a metal-walled Viton "O-ring" sealed vacuum chamber which contained an electron gun assembly and a water-cooled metal pedestal. Photographs of the equipment are shown in Figs. 6A-C, and a

sketch of the major parts is shown in Fig. 7. The sample surface temperature was observed through three quartz windows. Two of the windows shown in Fig. 7 were used for an automatic recording optical pyrometer ($0.65\text{-}\mu\text{m}$ wavelength) and a radiation pyrometer, respectively. The third window was used for visual observation. A vacuum between 10^{-5} and 10^{-6} torr was maintained by a large liquid-nitrogen-cooled trap placed in series with a 133-mm-diameter diffusion pump and a mechanical pump.

4.2 Electron Gun

As shown in Fig. 7, the electron gun consisted of a cathode assembly, anode ring, focus coil, deflection coil, and water-cooled copper and aluminum plates. The cathode was a 6.35-mm-diameter tantalum concentric-tube heater. The end surface of this heater served as an annular thermionic emitter in a

Fig. 5.

Time dependence of RTPR first-wall temperature and stress response to the energy fluxes given on Fig. 3. The time period during which the plasma internal energy W_{int} is uniformly deposited at the first wall is 2 s. Note that the burn period has been expanded at the left side of this figure.

Fig. 6A.

Photograph of TSEX apparatus showing EB gun, focusing coil, deflection coil, and sample holder.

Fig. 6B.
Edge view of EB gun.

Fig. 6C.
End view of EB cathode.

Pierce-gun configuration. Details of the EB-gun construction are shown in Fig. 8. The anode ring was fabricated from a fine-grained isotropic graphite (POCO HDP-1). Focusing of the EB was accomplished by a metal sheathed electromagnet of unknown manufacture. Deflection of the EB was accomplished with a Syntronic Instruments C2847 (specification Y123577) yoke assembly.

4.3 Power Supplies

The EB current was provided by a laboratory-constructed constant-current supply capable of furnishing a maximum of 1 A(dc) with a 10-kV compliance. This supply consisted of three-phase full-wave bridge rectifier driven from a variable autotransformer and followed by a series-pass current regulator in the form of a 10-kW tetrode transmitting tube. A feedback loop monitored the output current, compared it to a reference voltage and adjusted the transmitting-tube control-grid voltage accordingly. The cathode temperature was controlled by a regulated ac supply. Cathode-to-sample voltage was monitored and compared to a reference voltage, and the difference was applied to an amplifier and servomotor. The motor positioned the brush of a variable autotransformer, which supplied 60-Hz line power to a high-voltage filament

transformer. The output of this transformer was transmitted to the cathode heater through vacuum feedthrough connectors. The cathode assembly was held at a negative voltage with respect to the grounded sample chamber and sample. The anode ring was also grounded. The direct current to the focus coil was supplied by a regulated power supply, and the deflection coil was driven by a yoke amplifier which produced x- and y-output currents that were proportional to the corresponding input voltages.

Fig. 7.
Schematic representation of component arrangement in the TSEX apparatus.

Fig. 8.
Schematic diagram of EB gun.

Fig. 9.
Block diagram showing relationship between control elements of the TSEX apparatus.

4.4 Electron Beam Deflection Circuitry

The EB could be deflected in two orthogonal directions at frequencies up to ~ 100 kHz and amplitudes up to $\sim \pm 100$ mm by means of the above-described yoke amplifier and deflection coil. The circuit that provided the drive voltages to the amplifier consisted of three function generators, one oscillator and a laboratory-constructed control circuit. This circuit provided pulsed gain control of the x- and y-deflection circuit voltages. Figure 9 shows the electrical and control elements of the experiment, Fig. 10 shows the arrangement of the deflection circuit, and Fig. 11 gives the details of the laboratory-constructed control circuit.

In operation the electron beam was focused to a spot of about 10-mm diameter at the sample surface. The spot was a poorly defined image of the

Fig. 10.
Arrangement of elements of the EB deflection system.

circular cathode. The deflection circuit was driven with a 10-kHz sinusoidal voltage, such that the circuit in the y-deflection coil was 90° out of phase with respect to the current in the x-deflection coil. The combined deflection produced a circular motion of the beam about the axis of the sample. During the off-pulse period the diameter of the circular beam trajectory was about four times the sample diameter. To initiate a thermal pulse the application of a suitable signal to the control circuit reduced the EB trajectory diameter to about one-half of the sample diameter. The net effect on the sample was to deposit energy in a circular pattern with a radial but not circumferential nonuniformity. With slightly more energy deposited at the periphery than at the center, the tendency of the sample to buckle from thermal stresses was reduced, and a relatively uniform temperature distribution (radially) was produced at the vacuum- Al_2O_3 interface.

4.5 Diagnostic Instrumentation

Sample surface temperature from two pyrometers (Fig. 7) and the pulse voltage applied to the deflection circuits (which indicated the duration and repetition rate of the EB impingement on the sample) were recorded. A Leeds and Northrup (Mark I Model 6841) precision automatic optical pyrometer (0.65- μm wavelength) was used to measure continuously the sample surface temperature. The specified 1-s response time for following to 99% of a step change in temperature was too slow for accurate recording of the temperature transient with the optical pyrometer, however, and a radiation pyrometer was constructed by modifying a Leeds and Northrup Lens Spectray 65, (Model 8886). The modification shown in Fig. 12 consisted of replacing the detector with a Texas Instrument LS400 photodiode and external amplifier. Both pyrometers were calibrated against a type-K (Chromel-Alumel) thermocouple that was spot welded to the back of a sample. Output from the Mark I pyrometer was recorded on a two-pen Hewlett Packard Model 7100B strip chart recorder using Model 17501A amplifiers, and the pulse voltage was recorded on the remaining channel. The response time of the recorder was greater than that of the pyrometer but not fast enough to follow the temperature excursion rise time. The output of the radiation pyrometer was recorded on a Mosley Model 7700 thermal-pen recorder using a Model 8809A signal coupler. The response time of this recorder was faster than 0.1 s for full-scale deflection, while the response time of the LS400 photodiode circuit was a few milliseconds.

5. EXPERIMENTAL PROCEDURE

5.1 Sample Preparation

Samples were obtained from the materials technology group (CMB-6). Disks were punched from 1-mm-thick rolled sheet of Nb-1Zr alloy and coated with Al_2O_3 using a plasma-arc-spraying process. The Al_2O_3 coating was ~ 0.3 mm thick. Samples in this condition could be EB-heated directly, but a thin electrically conducting coat of graphite or metal (Pt) produced more reliable results.* A ~ 10 - μm -thick platinum layer was applied by physical vapor deposition in the TSEX apparatus. A second series of $(0.3\text{-mm})\text{Al}_2\text{O}_3/(1.0\text{-mm})$ Nb-1Zr samples, which had been sputter coated with niobium, was obtained from the materials technology group. The test specimens were manually ground flat at the Nb-1Zr surface with 600 grit metallographic paper. One of the thermally pulsed samples (Sample 10) had the edge ground on a dry alumina wheel before the platinum coating was applied.

5.2 Pyrometer Calibration

Prior to thermal pulsing a Pt-coated $\text{Al}_2\text{O}_3/\text{Nb-1Zr}$ sample was suspended in vacuo 20 mm above the sample pedestal (Fig. 7) by a type-K thermocouple that had been spot welded to the center of the niobium surface. The EB cathode was heated to the same temperature used during the pulse testing, as indicated by the magnitude of the ac heating current supplied through the filament transformer. Accelerating voltage was applied to the gun, and the deflection circuit was adjusted to provide a continuous and diffuse EB bombardment of the platinum-coated Al_2O_3 surface of the sample. Both pyrometer outputs were then recorded as functions of the thermocouple output at a series of sample temperatures between 1000 and 1300 K. The temperature gradients through the sample were assumed to be negligible in this case, this assumption being verified by calculation. The bright EB cathode (~ 2500 K) illuminated the sample, and the reflected light contributed to the pyrometer output. This

*Without a thin conducting coating on the Al_2O_3 surface a corona discharge would form at the sample if the EB current exceeded ~ 200 mA at ~ 1 kV when the sample temperature was ~ 800 K. The coatings used were chemically inert and thin enough to have no structural mechanical effects. These thin metallic coatings also proved to be valuable in subsequent flash thermal diffusivity measurements (Sec. 6.3).

calibration was repeated after the thermal pulse tests at the same EB-cathode heating current.

5.3 Thermal Pulse Test Procedure

A thermal pulse test consisted of placing a sample on the water-cooled pedestal, evacuating the chamber, energizing the EB gun and control circuitry, and recording the pyrometer and deflection circuit outputs as a function of time. Sample temperature was a function of EB accelerating voltage, length of time the EB bombarded the sample, and pulse repetition rate, provided the thermal coupling of the test specimen to the pedestal remained constant. Suitable temperature excursions were produced by adjusting these electrical and temporal parameters, and temperature uniformity across the surface of the specimen was achieved by adjusting the deflection and focus circuitry. Typically, an 8-kV, 400-mA EB directed onto the test specimen for 200 ms once every 6.5 s resulted in a constant base-line surface (Al_2O_3 /vacuum) temperature

Fig. 11.
Circuit diagram of EB deflection control.

Fig. 12.
Circuit diagrams of radiation pyrometer.

of ~ 1000 K and a thermal excursion relative to this base-line temperature of ~ 200 - 300 K. When a sample was subjected to many (> 100) pulses, the deflection coils (Fig. 7) would be radiatively heated by the hot test specimen, and the resulting change in EB trajectory would cause a drift in the sample energy density. This problem can be readily solved by redesign (i.e., use of radiation shields). The EB and sample parameters given above are typical for this experiment.

6. EXPERIMENTAL RESULTS

6.1 Thermal Response of Test Specimens

It is emphasized here that the goal of these "proof-of-principle" tests is to demonstrate the feasibility of using programmable pulsed EB irradiations to simulate reactor-like thermal excursions for insulator-metal first-wall composites. Little control was given to either sample fabrication, preparation, or thermal history. A total of 16 test specimens were available, only two actually being used for extensive testing in the TSEX apparatus. Table I summarizes both the condition of these test specimens and the results of flash thermal diffusivity measurements made subsequent to the TSEX tests. Sample 10 received the greatest number of thermal cycles (~ 800) in two separate runs and over a range of thermal (i.e., EB) conditions. Table II summarizes the thermal and mechanical history of this sample. Figure 13A shows the radiation pyrometer trace from several of the 120 pulses described in History (F) of Table II (120 pulses), the flat top representing the limit of recorder pen travel. Shown also on Fig. 13B is the calibration curve for the radiation pyrometer. The corresponding output from the optical pyrometer and the pulse applied to the control circuit are shown on Fig. 14; the initiation of both optical pyrometer and control pulses occur at the same time, the offset shown on Fig. 14 being purely mechanical to permit cross-over of the recorder pens. Although the optical pyrometer gave an accurate recording of the steady-state base-line temperature, it was incapable of following the rapid thermal excursion; the oscillation shown on Fig. 14 represents an instrumental overshoot. The agreement between the base-line temperatures as measured by the optical pyrometer and the thermocouple-calibrated radiation pyrometer generally was good throughout all runs.

Although the diagnostic results from this experiment are inadequate to resolve fully by calculation the complete thermal and stress history of a

TABLE I

SUMMARY OF TEST SPECIMEN CONDITIONS AND
THERMAL DIFFUSIVITY MEASUREMENTS

Sample	Condition	Metal Thickness (mm)	Coating Thickness (mm)	(e) $t_{\frac{1}{2}}$ (ms)	Measured composite thermal diffusivity (cm ² /s)
1	uncoated Nb-1Zr	1.01	—	—	—
2	"	0.99	—	6	0.230
3	"	0.96	—	—	—
4 ^{a,d}	Al ₂ O ₃ coated	1.01	0.30	26	0.092
4 ^a	Al ₂ O ₃ coated, Pt deposit	1.01	0.30	50	0.048
7 ^{a,b}	Al ₂ O ₃ coated, Pt deposit	0.96	0.30	36	0.061
8 ^{a,b}	Al ₂ O ₃ coated	1.00	0.30	25	0.094
8 ^{a,b}	Al ₂ O ₃ coated, Pt deposit	1.00	0.30	37	0.063
9 ^{a,b}	Al ₂ O ₃ coated	0.98	0.30	26	0.087
9	Al ₂ O ₃ coated, Pt deposit	0.98	0.30	34	0.066
10 ^{a,b}	Al ₂ O ₃ coated, Pt deposit (thermally pulsed)	0.92	0.30	35	0.059
11	Al ₂ O ₃ coated, Nb deposit	1.03	0.28	50	0.047
12	"	1.07	0.48	84	0.040
13 ^c	"	1.03	0.41	45	0.064
14	"	1.05	0.46	85	0.037
15	"	1.02	0.33	42	0.056
16	"	1.03	0.43	59	0.034

^aSamples 4 through 10 thicknesses were determined from total thickness and assumed uniform coating thickness.

^bOnly Samples 7 and 10 experienced significant exposure in the TSEX apparatus, and of these, exposure histories were recorded only for Sample 10.

^cThis sample produced an unreliable oscilloscope trace in diffusivity apparatus.

^dSamples 4, 8, and 9 show higher values of diffusivity than those that have either a platinum or niobium surface coating on the Al₂O₃ layer. This behavior is to be expected, since the coating is translucent, and the incident energy is partially deposited within this layer when no optically dense surface film is present.

^eHalf-time for thermal rise in thermal diffusivity measurement.

TABLE II
MECHANICAL AND THERMAL HISTORY OF
(0.3-mm) Al₂O₃ / (1.0-mm) Nb-1Zr SAMPLE 10 (RE: TABLE I)

Mechanical History: Edge ground on a dry Al₂O₃ wheel to produce greater (electrically) conducting edge area.

Thermal History:

- A. Pt-coated for 20 minutes in TSEX apparatus.
- B. Placed in water-cooled Cu pedestal in TSEX apparatus to perform pyrometer calibration up to 1189 K.
- C. Subject sample to thermal cycle under a range of EB conditions shown below.

EB Voltage (kV)	EB Current (mA)	Power Density ^a (W/cm ²)	Minimum Surface Temperature (K)	Maximum Surface Temperature (K)	Temperature Excursion (K)	Number of Pulses
8	400	632	1023	1258	235	20
8	360	569	1028	1273	245	20
8	340	537	1013	1225	212	10
8 _b	340	537	1023	1250	227	10
8 ^b	320	506	993	1176	183	20
8	450	711	1023	1273	250	400

^aBased upon a total focused beam area equal to that of the sample (5.07 cm²). Because of the EB diffusiveness, this area may be considerably larger.

^bFor this case the sample was repeatedly pulsed once every 6.5 s with 200-ms-long pulses.

- D. Sample was removed, bottom was slightly convex, bottom was then flattened with #600 grit paper, sample was replaced into TSEX apparatus.
- E. Third temperature calibration was made up to 1228 K with a 450-mA, 8-kV EB. The EB was deflected in a circle with gradually reduced radius to vary the sample temperature without changing the EB gun cathode brightness.
- F. Subject sample to thermal cycle under a range of EB conditions shown below:

EB Voltage (kV)	EB Current (mA)	Power Density ^a (W/cm ²)	Minimum Surface Temperature (K)	Maximum Surface Temperature (K)	Temperature Excursion (K)	Number of Pulses
8	450	711	1013	1138	325	120
8	450	711	1013	1338-1123 ^b	325-110 ^b	80
8	450	711	1013	1123-1073 ^b	110-60 ^b	200

^aBased upon a total focused beam area equal to that of the sample (5.07 cm²). Because of the EB diffusiveness, this area may be considerably larger.

^bBecause of radiant heating of focusing coil, the EB energy onto the sample and, hence, the sample temperature, drifted with time.

- G. Sample removed from TSEX apparatus, type-K thermocouple spot welded to underside of sample and fourth temperature calibration made up to 1330 K.

Fig. 13A.
Radiation pyrometer output for a series of pulses at conditions described in Table II under Sample 10 History (F), 120 pulses.

Fig. 13B.
Temperature calibration of radiation pyrometer.

Fig. 14.

Optical pyrometer and pulse control signals for the conditions described in Table II under Sample 10 History (F), 120 pulses. Although the type-K thermocouple calibrated optical pyrometer gave good temperature readings for both the base-line and peak temperatures, the optical pyrometer was not capable of following the rapid thermal excursion (compared to radiation pyrometer response, Fig. 13B).

given sample, the heat transfer code AYER⁹ was used in conjunction with the known surface temperature excursion, (approximate) heat fluxes, and pulse duration rate to estimate the sample response. Figures 15A and 15B show the results of these computations. In Fig. 15A is given an approximate match to the conditions summarized under thermal History (C) of Table II. For this case the temperature-dependent thermophysical properties of pure Al_2O_3 and Nb were used,¹⁰ and for the stated pulse duration and repetition rate the pulsed heat flux F_0 (W/cm^2) was varied until the base-line and peak surface (Al_2O_3) temperatures simultaneously fell within the range of the TSEX results (Table II). The emissivity of both Al_2O_3 and Nb-1Zr was fixed at 0.6, and the heat loss was assumed to occur solely by radiation from both surfaces of the sample. The resultant heat flux F_0 computed to fall onto the sample was about 50% that given on Table II, which assumes all of the EB current impinged on

Fig. 15A.
Computed temperature and stress responses of TSEX sample to conditions similar to those listed under History (C) in Table II.

Fig. 15B.
Computed temperature and stress response of TSEX sample to conditions similar to those listed under History (F), 120 pulses, in Table II. Experimental surface temperature data (Fig. 13) are also shown for comparison.

the sample. Increasing either F_0 or the heat loss from the sample (i.e., combined conduction and radiation of the Nb-12r surface) would not permit simultaneous matching of the measured base-line and peak surface temperatures. For the given conditions the computed thermal stress transient at both Al_2O_3 surfaces as well as the net interfacial shear (debonding) stress are also summarized on Fig. 15A. Compared to Fig. 5, the computed TSEX stresses are about 50% greater than those anticipated for the RTPR. No attempt was made to duplicate the actual thermal and stress waveform expected for the RTPR, although the easily programmed EB used in TSEX will permit simulation of all aspects of the RTPR burn cycle except the rapid cooling phase that occurs at the end of the plasma-dump phase (Fig. 5).

Using the radiation pyrometer trace and associated calibration curve given on Fig. 13 it is possible to determine an actual temperature trace for the 120 pulse condition given for Sample 10 under History (F) on Table II. The time dependence of the experimentally determined Al_2O_3 surface temperature, as well as the results of the AYER heat transfer calculation, are given on Fig 15B. The computed temperature and stress transient apply here for an emissivity of 0.75 and $F_0 = 425 \text{ W/cm}^2$, which again is $\sim 50\%$ below the pulsed heat load given on Table II (History F). To match better the experimental base-line and maximum temperature, F_0 was decreased to 405 W/cm^2 , and the density of Al_2O_3 was decreased to 85% of the theoretical value (more in agreement with the plasma-arc-sprayed material). Again, the temperature-dependent thermophysical properties of Al_2O_3 and Nb were used by the AYER computation. Within the accuracy limits offered by the radiation pyrometer calibration, the assumed emissivities for both Al_2O_3 and Nb, the thermophysical properties used, and the assumed boundary condition at the Nb-vacuum interface, the agreement shown on Fig. 15B is considered good. Although better agreement between the calculated and experimental surface temperature curves is possible by further adjustment of relevant parameters, the computed stress transient shown on Fig. 15B is relatively insensitive to such adjustments. Compared to Fig. 5, these TSEX-generated stresses shown in Fig. 15B are ~ 3 times that expected for the RTPR first wall.

The uncertainty in the thermophysical properties of the plasma-arc-sprayed Al_2O_3 used in these experiments is great. Section 6.3 addresses this problem within the context of experimental results from thermal diffusivity measurements.

6.2 Mechanical Effects in the Test Specimens

For the most part, using both optical and scanning electron micrographic (SEM) methods, no visible difference could be detected between the highly exposed specimen (Sample 10) and unexposed control samples. The microscopy results given here, therefore, pertain only to Sample 10 after the thermal history summarized in Table II had been achieved. The thickness of the Pt coating applied to the exposed Al_2O_3 layer is shown in Fig. 16 and is seen to be less than $1\text{ }\mu\text{m}$. Figure 16 also shows the morphology of a typical $\text{Al}_2\text{O}_3/\text{Nb-1Zr}$ interface, using both optical and SEM techniques. The jagged and interlocking nature of this interface undoubtedly resulted in the observed resistance to gross delamination under severe thermal cycling. The multiple phases observed in the Al_2O_3 are shown in Fig. 17. This multiphase character appeared to result from the plasma-arc-spraying process and is not a result of thermal cycling. At least three Al_2O_3 phases are seen, although an unambiguous identification of each was not made. X-ray examination of the coating revealed that the major crystalline phase was (cubic) $\gamma\text{-Al}_2\text{O}_3$ with (hexagonal) α -alumina present as a minor constituent. Another major phase could best be described as a highly distorted, nearly amorphous phase similar to the η -form of Al_2O_3 . Seen also in the typical photographs depicted in Figs. 16-17 is an appreciable microcrack structure, which is resolved in more detail by SEM in Fig. 18. This internal crack structure appears to be identical in both thermally cycled and control specimens, and the degree of crack extension induced by cyclic thermal stresses could not be determined.

The defects so far described can be considered typical in both test and control samples. Typical defects are shown in Figs. 19 and 20. The results of an unintentional, local melting of the Al_2O_3 coating by an inadvertent EB overfocusing are shown in Fig. 19. Although the Al_2O_3 resolidified intact, a crack was generated parallel to the substrate surface. Figure 20 shows two edge delaminations observed visually in Sample 10 after the thermal pulse history summarized in Table II. Details of this particular crack structure are also depicted in Fig. 21. Several cracks or delaminations in the Al_2O_3 layer at the sample periphery are observed, and one crack appeared to be a delamination combined with a radial surface crack that extended from the edge inward toward the center for about 2 mm. No other external evidence of change could be detected. The radial crack appears to start at the $\text{Al}_2\text{O}_3/\text{Nb-1Zr}$ interface and extends to the Al_2O_3 surface after a radial traverse of

~1.3 mm. Although this crack was definitely induced by the exposure summarized in Table II, the edge location and the pre-exposure edge grinding that this particular sample experienced preclude the attachment of special significance to this single defect. More control of sample fabrication, thermal history, and geometry must be exercised before the origin and significance of each unique defect can be ascertained. Generally, the bond integrity, microcrack structure, and morphology of the sample showed no detectable change after experiencing the thermal history summarized in Table II.

Fig. 16.

Optical and scanning electron microscopy (SEM) photographs showing the Al₂O₃/Nb-1Zr bond integrity (Sample 10).

Fig. 17.

Optical and scanning electron microscopy (SEM) photographs showing the three unidentified phases in the plasma-arc-sprayed Al₂O₃ (Sample 10).

Fig. 18.
Scanning electron microscopy (SEM)
photographs showing typical internal
crack structural within the Al_2O_3
layer (Sample 10).

Fig. 19.
Optical micrograph photograph of a region of sample where
local melting of Al_2O_3 occurred because of EB over-
focusing.

Fig. 20.
Edge defects from thermal pulsing
(Sample 10). Specimen had to be edge
ground prior to thermal cycle.

Fig. 21.
Edge defects from thermal pulsing (Sample 10). Specimen
had been edge ground prior to thermal cycle.

6.3 Thermal Diffusivity Measurements

In an effort to monitor latent and/or unresolved effects of Al_2O_3 delamination on the heat transport characteristics of the (0.3-mm) Al_2O_3 /(1.0-mm)Nb-1Zr composite, the thermal diffusivity for all specimens was measured. The thermal diffusivity measurements were made using a xenon flash-lamp apparatus. The method consists of observing the output of a thermocouple pressed against the back (Nb-1Zr) face of the room-temperature sample while the front (Al_2O_3) face was exposed to a pulse of radiant energy from the flash lamp. The results for a variety of samples are listed in Table I. The thermal diffusivity of Sample 10 is not significantly different from the other samples, although enough variation from sample to sample exists to obscure small differences. The interesting feature of these results is the low average value of diffusivity for most of the composite samples. The low measured composite diffusivity requires that either the diffusivity of the Al_2O_3 layer be very low, the thermal resistance of the interface between the Al_2O_3 and Nb-1Zr alloy be very high, or that both effects be operative. To test the accuracy of the thermal diffusivity measurements, Sample 7 (Table I) was re-examined in both the forward and reversed direction, and an Armco iron reference sample was measured. Table III summarizes the results from these measurements along with the Nb-1Zr (Sample 2, Table I) data, and agreement is good. Table IV summarizes the literature values of the relevant thermophysical properties of Al_2O_3 and Nb.

By postulating a low thermal resistance at the coating-metal interface, a numerical solution for two layered composites¹¹ can be employed to compute the thermal diffusivity of the alumina layer.¹² The following values (Table IV) were used for the thermal properties used in these calculations:

- (1) Absolute heat capacity of coating relative to metal = 0.35, allowing for 86% of theoretical density for the alumina layer,
- (2) Thermal diffusivity of the metal, (Nb-1Zr) equals $0.23 \text{ cm}^2/\text{s}$,
- (3) The modified Fourier number = $\alpha_{\text{Nb}} t_{\frac{1}{2}} / \ell^2_{\text{Nb}} = 0.828$, and
- (4) $(\ell^2/\alpha)_{\text{Al}_2\text{O}_3} / (\ell^2/\alpha)_{\text{Nb}} = 1.4$ where α = diffusivity, $t_{\frac{1}{2}}$ is the half-time of the temperature rise at the metal (back) surface, and ℓ is the layer thickness.

This calculation yielded an Al_2O_3 diffusivity of $0.015 \text{ cm}^2/\text{s}$, which compares favorably with the range of 0.014 to $0.017 \text{ cm}^2/\text{s}$ obtained from plasma-arc-sprayed alumina (Table III) without a substrate present, but is a factor of 8.1 less than the literature values reported for the room-temperature properties of α -alumina (Table IV).

This large difference in thermal diffusivity of the coating from the accepted value for α -alumina probably results from one or more of the following conditions:

TABLE III

SUMMARY OF ROOM-TEMPERATURE THERMAL DIFFUSIVITY MEASUREMENTS

Sample	Thermal Diffusivity (cm^2/s)
$\text{Al}_2\text{O}_3/\text{Nb-1Zr}$ Sample 7 with metal facing flash lamp	0.060
$\text{Al}_2\text{O}_3/\text{Nb-1Zr}$ Sample 7 with ceramic facing flash lamp	0.062
LASL Armco iron reference sample	0.206
Accepted value for Armco iron	0.199
Nb-1Zr Sample 2	0.230
Nb literature value	0.235
Plasma-arc-sprayed Al_2O_3	0.014-0.017

^aUncertainty is a result of variable thickness of sample.

TABLE IV

ROOM-TEMPERATURE THERMOPHYSICAL PROPERTIES¹⁰
 α -ALUMINA^a and NIOBIUM

	Al_2O_3	Nb
Density (g/cm^3)	3.80	8.55
Heat capacity c ($\text{J}/\text{g K}$)	0.79	0.26
Thermal conductivity k ($\text{W}/\text{cm K}$)	0.53	0.35
Thermal diffusivity α (cm^2/s) = $k/c_p\rho$	0.12	0.24
Cross-sectional area a (cm^2)	5.07	5.07
Thickness ℓ (cm)	0.03	0.10
Absolute heat capacity H (J/K) = $c_p \rho a \ell$	0.46	1.13

^aFor $T > 600 \text{ K}$ the following expressions are used for the thermophysical properties of Al_2O_3 , where T is the temperature in Kelvin units.

$$\begin{aligned}
 k (\text{W}/\text{cm K}) &= 309./T^{1.20} \\
 c_p (\text{J}/\text{g K}) &= 0.4184 T^{0.18} \\
 \alpha (\text{cm}^2/\text{s}) &= 194/T^{1.38}
 \end{aligned}$$

- (1) Possibly lower thermal transport properties for the two phases other than α -alumina in the coating,
- (2) The defect morphology in the coating as indicated by the optical and SEM photomicrographs.
- (3) A high thermal resistance of the coating-metal interface.
- (4) A strong influence of coating porosity which could have been as high as 15%.

Of these possibilities it would appear that the defect structure and the thermal properties of the three phases in the coating would be most effective in reducing the thermal diffusivity. The presence of a major phase of poorly crystalline material could be an important factor and one which may be modified by changes in the plasma-arc-spraying technique.

In order to gain more insight into the cause(s) for the abnormally low room-temperature thermal diffusivities measured for the plasma-arc-sprayed Al_2O_3 , a chip of this material was annealed at 1170 K in air for 24 hours, after which it was pulverized and a Debye-Scherrer pattern obtained. This pattern was similar to those previously noted, showing broadened and weak lines from α - Al_2O_3 , γ - Al_2O_3 , and probably at least one other metastable Al_2O_3 phase. The Al_2O_3 chip was next heated in vacuo at 2090 K for 1 hour and a Debye-Scherrer pattern gave strong, sharp α - Al_2O_3 reflections and weak lines from the other phases. The sample color had also changed from the initial dull gray to bright white. An additional anneal at 2190 K for 1.5 hours showed no further change in the sharpened Debye-Scherrer pattern. An $\text{Al}_2\text{O}_3/\text{Nb-12r}$ thermal diffusivity sample was annealed for 1.5 hours at 2190 K, after which the diffusivity was measured. This sample (physically the same one that had been measured previously, Table III last entry) gave a diffusivity value of $0.11 \text{ cm}^2/\text{s}$. Although this value may lack precision because of variations in sample thickness and because of the short half-rise time (2.5 ms), it is clearly distinguishable from the previous result ($0.015 \text{ cm}^2/\text{s}$). These findings give strong indications that the low room-temperature diffusivity measured for plasma-arc-sprayed Al_2O_3 on Nb-12r is caused by the morphology of the Al_2O_3 phase rather than by unresolved interfacial thermal resistance.

Evaluating the expression for the thermal diffusivity (Table IV) at the temperature of the experiment ($T \approx 1000$ K) gives $\alpha = 0.01 \text{ cm}^2/\text{s}$ for pure Al_2O_3 . The conjecture is made that the defect structure in room-temperature plasma-arc-sprayed Al_2O_3 may represent a high-temperature form which has been "frozen" into the materials used in these studies. The relatively good agreement between the calculated thermal transient (which used the temperature-dependent thermophysical properties of Al_2O_3) and the experimentally measured transient (Fig. 15B) tends to substantiate this hypothesis. Considerably more work is required to resolve and to control the defect structure of plasma-arc-sprayed Al_2O_3 , particularly with respect to the temperature dependence of both structural and thermophysical properties.

7. SUMMARY AND CONCLUSIONS

The results of this experiment indicate that programmed EB heating of sample first-wall configurations represents a promising and useful test of thermal/mechanical durability of layered, composite systems. Temperature excursions predicted in reactor design studies can be achieved with convenient control over the parameters that determine the energy input and removal. The sample size is small enough to be economical to fabricate, and sample handling in the apparatus is minimal. The ease and accuracy of the sample temperature measurement is satisfactory, and the stability of the system is sufficient for tests to be conducted with thousands of thermal pulses. The fact that some thermally produced defects were observed after a test of about 800 pulses (Sample 10) suggests that the method would provide a rapid means of comparing samples of different materials, configurations, and pretest defect structure. The components in the EB gun and vacuum chamber are relatively inexpensive and could be readily replaced if contaminated by irradiated samples. Once constructed and tested, the apparatus could be operated by technician-level personnel with minimum technical supervision. The construction of an apparatus which would be dedicated to thermal pulse testing of first-wall composites would include the following changes in the apparatus used in this "proof-of-principle" experiment:

- Separate electron gun chamber that can be isolated from the sample chamber.
- Redesign of EB-gun cathode to a larger ring type, as described by Harries,¹³ to avoid a direct optical path from the hot cathode to the

sample.

- Fabrication of a simple deflection circuit to replace the expensive commercial units assembled for this test.
- Construction of a sample pedestal with a spring-loaded miniature thermocouple to record the metal surface temperature of the sample. The sample and pedestal would be electrically isolated from ground so that the EB current intercepted by the sample could be recorded.
- Possible redesign of the pedestal to provide a more controllable and efficient heat sink which operates at a higher temperature. A thin molten lead film was tested with good success in improving heat transfer between the sample and the water-cooled plate. Similar arrangements would permit higher EB energies, more energy input, and a faster cool-down between heating pulses, thereby more accurately simulating the post-burn cooled-down phase of the RTPR power cycle (Fig. 5).

Although this experiment was not intended to provide controlled thermal-shock testing of first-wall composites, the limited sample exposures that resulted from these "proof-of-principle" tests have given some indication of the toughness of the $\text{Al}_2\text{O}_3/\text{Nb-1Zr}$ system. Not only did this material survive with little or no mechanical damage after a large number of reactor-like thermal transients, but these samples have experienced a significant number of room-temperature to operating-temperature (~ 1000 K) cycles throughout the TSEX check-out. Under some circumstances the EB was inadvertently focused onto a test specimen to such an extent that local melting of the Al_2O_3 insulator occurred without observable insulator-metal debonding at or around the melted zone.

Although the question of thermally induced changes in the defect structure and thermophysical properties of Al_2O_3 has been raised by these tests, the control exercised in both sample fabrication and history was not sufficient to draw firm conclusions. The observed defects and deviations in thermophysical properties undoubtedly are a result of the plasma-arc-spray process rather than being caused by the exposure to thermal cycle. Future experiments which incorporate the above mentioned innovations and/or changes should address this question in considerably more detail.

Other design changes in the TSEX apparatus should be noted, although these changes will require a developmental effort. For example, in situ measurement of the sample stresses and thermal diffusivity would provide a powerful and convenient diagnostic tool. An indication of the thermal stresses can be obtained by use of either high-temperature transponders or the measurement of sample deflection when placed in a specific configuration. The continuous measurement of the thermal diffusivity would allow changes in sample defect structure and/or bond integrity to be monitored as a function of number of thermal cycles without cooling the sample to room temperature. Lastly and most importantly, the ceramic-metal composite envisaged for the RTPR must periodically function under pulsed dielectric stress in addition to surviving a large number of thermal mechanical excursions. Ultimately, therefore, the dielectric integrity of the test specimen will have to be tested as a function of thermal history in order to ascertain when and by which mechanism the dielectric strength has degraded to a point where use as a first-wall material in a pulsed, high-beta fusion reactor no longer is operational.

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