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FATIGUE TESTING OF PLASMA-SPRAYED THERMAL BARRIER COATINGS

VOLUME 2

By

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FINAL REPORT

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1. Acknowledgments

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2 Plasma-Sprayed Bend Fatigue Testing

In addition to conducting the compression and tensile test program on the Pratt & Whitney EB-PVD coating (Volume 1 of this report), the cooperative agreement also required SwRI to assist Caterpillar Inc. in its developmental effort of a plasma-sprayed thermal barrier coating for diesel engines by providing test support. SwRI's task consisted of the fatigue screening of candidate thermal barrier coating materials and the generation of a data base for the selected candidate material.

2.1 Specimen Configurations

Bending fatigue specimens were supplied by Caterpillar Inc. in three different formats:

1. 0.75-inch X 4.75-inch substrates with coatings,
2. 0.38-inch X 3.00-inch substrates with coatings,
3. 0.375-inch X 2.15 X 0.085-inch average thickness coating material only (no substrates).

Respective thickness values of the specimens are reported in the tables detailing the test matrices.

2.2 Test Setup

Both the screening and the data base test matrices were conducted in four-point bending mode. The test fixtures were fabricated from 316 stainless steel and were configured as shown in Figure 1[1]. The inner span of the fixture was fastened to the water-cooled, upper extension column, which in turn was attached to the stationary load cell of an 11-KIP MTS hydraulic test system. The outer span was supported by a stainless steel sphere, which was held on the centerline of the load train by a conical bore in the lower, water-cooled extension column. Application of the stainless steel sphere provided the necessary final alignment at the fixture-specimen contact points. The lower extension column was attached to the hydraulic actuator shaft of the testing system. A lower capacity, more sensitive load cell was inserted between the system's load cell and the upper extension column for tests requiring small amplitude loads; namely, for the 426-B test group.

Two load span configurations were used in the program, 40-mm (1.575-inch) and 80-mm (3.15-inch) total distance between outer load points, depending on the specimen configuration.

In the elevated temperature tests, heating of the specimens and test fixtures was accomplished with a lightweight, split, clam-shell furnace shown in the figure. The hot zone temperature of the furnace was controlled with a "K" type thermocouple positioned in the approximate center of the hot zone. Lightweight thermocouples, attached to the bend fixture components, were used to monitor the fixture temperatures to assure that thermal stability of the test system was achieved. It was assumed that the specimen was at the desired test temperature when all test fixture components reached thermal stability at that temperature.

2.3 Specimen Preparation

In the initial stage of the screening program, specimens were tested in the "as received" condition. After several consecutive substrate failures, it was determined that premature fatigue crack initiation was taking place at the sharp edges of the substrates or at surface defects on the face of the substrates.

[1] Figures and tables are in Section 3.

A surface preparation procedure was instituted which included rounding of the sharp substrate corners and polishing of the face of the substrates in steps to a final 1 micron finish. Use of this procedure extended the life of the substrates to the point where generation of coating failure became possible.

Although the polishing procedure greatly improved substrate performance, substrate failure remained a continuously bothersome problem. In order to further improve substrate performance, an attempt was made to shift the neutral plane of bending of the "composite" (substrate/coating) beam in the direction of the free substrate surface and thereby decrease stress amplitudes at the free surface of the substrate, where the fatigue failures occurred. This shift can be accomplished by reducing the substrate thickness. Removal of material from the substrate surface was accomplished using surface grinding procedures and post-grinding polishing. Disappointingly, reduction of the substrate thickness did not produce the anticipated improvements.

2.4 Test Matrix

A total of 97 specimens were tested in the program: 35 in the screening phase and 62 to generate a data base. Test specimen designations, along with respective specimen dimensions, fatigue load amplitudes, and failure data, are reported in Tables 1 through 8.

A compressive bending fatigue loading mode was specified by Caterpillar for both screening and data base tests. Six candidate materials were tested, as shown in the tables. For the data base generation, both tensile and compressive bending modes were utilized.

2.5 Test Procedure

The four-point bending fixture described above was utilized for both tensile and compressive bending modes. The difference between the two procedures was determined by the specimen orientation in the test fixtures. Tensile mode implies that the surface stresses in the coating on the specimen were of a tensile nature.

All screening tests were performed at an "R" ratio of 0.07, and the specimens were fatigued to failure without any additional measurements.

In the data base test matrix, a total of 17 specimens were designated for static bend fracture testing. The specimens designated for room temperature fracture tests were instrumented with strain gages to determine the static, elastic modulus values for the coating. Head displacement values were measured simultaneously with the strain gage outputs to obtain a compliance value for the load train. Since application of strain gages at 400° C was not feasible, measured head displacement values along with the previously determined compliance values were used to calculate the elastic modulus for the coatings. All static fracture tests were performed at a constant, 0.050-inch per minute, head displacement rate.

Fatigue testing was conducted at 0.07 and 0.60 "R" ratios and at the maximum frequency rate obtainable for the particular specimen group. It was determined early in the test program that variation in cyclic frequency had no detectable effect on the fatigue strength levels. Fatigue frequencies ranged between 1 and 15 Hz for the program.

In addition to substrate failures, ceramic coating failures due to high contact stresses were also a problem. Attempts to reduce the effects of the high contact stresses through the use of copper or aluminum "load spreader pads" were mostly unsuccessful, and the approach was abandoned.

Observations regarding individual specimen failure modes are presented in Tables 3 through 8.

The second, major objective of this test program was to generate spalling-type failures observed in the actual engine tests. This objective was met with only partial success; only four failures of this type were obtained during the entire test program. It became clear that the substrate/coating specimen configuration tested in four-point bending mode was not the ideal experimental approach for generating this type of failure. After the desired data base information was obtained, further attempts to generate spalling-type coating failures were abandoned. It is suggested that alternative approaches, such as pre-compressed rotating beam fatigue testing, be investigated for producing such data.

2.6 Data Analysis

Test data analysis was performed entirely by Caterpillar personnel. Test results from individual tests were transmitted as they were generated, allowing Caterpillar personnel to select the most appropriate test parameters for the test to follow.

Partial test results generated during the program were presented by Randolph C. Brink of Caterpillar Inc. in the article, "Material Property Evaluation of Thick Thermal Barrier Coating Systems," published in Transactions of the ASME, Journal of Engineering for Gas Turbines and Power, Volume 111, Number 3, July 1989, pp. 570-577.

3 Figures and Tables

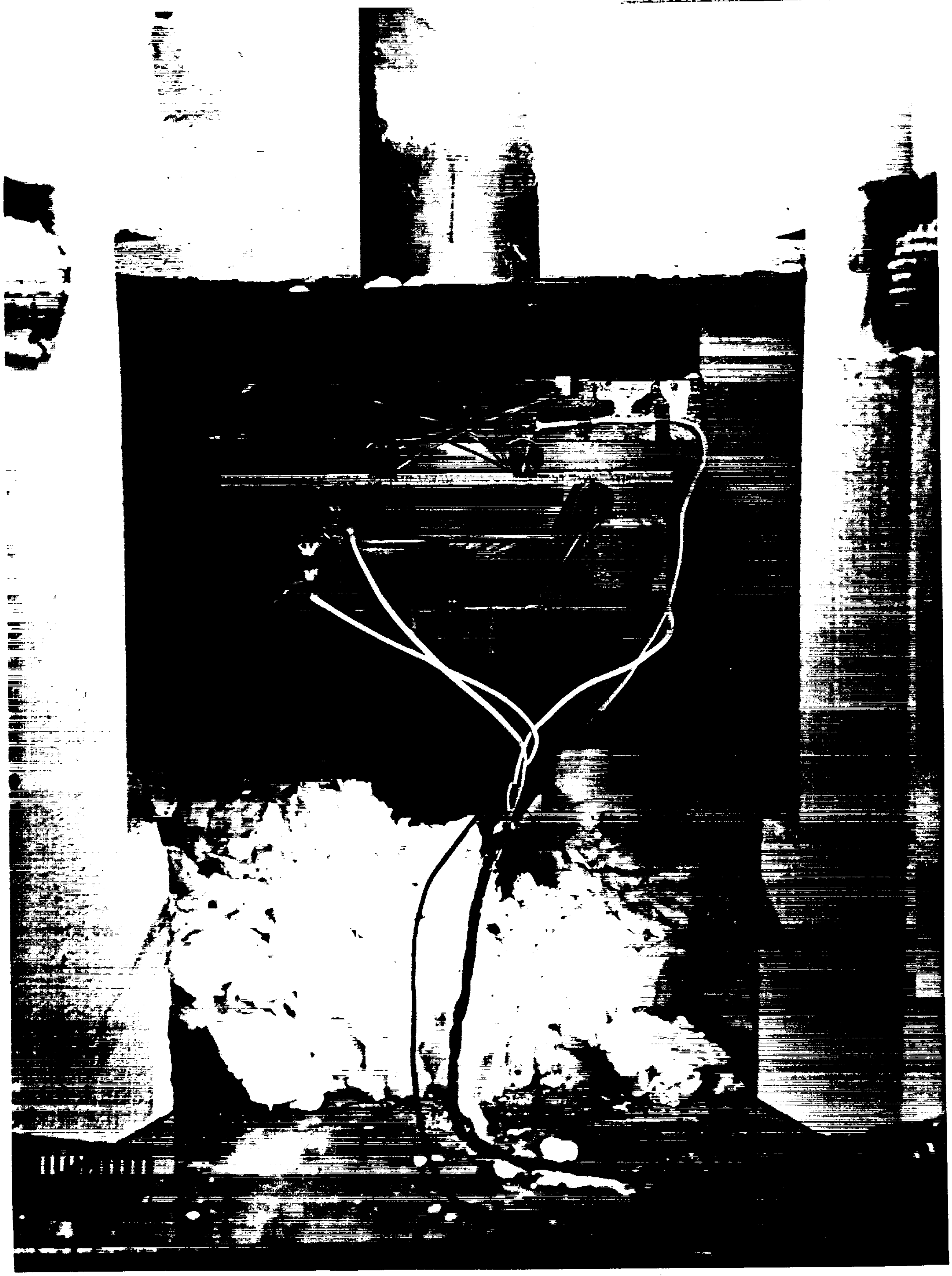

FIGURE 1. ELEVATED TEMPERATURE, FOUR-POINT BEND TEST SETUP

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

TABLE I

SCREENING TEST SPECIMEN DIMENSIONS

Specimen Number	Thickness (Inch)		
	Center	End #1	End #2
377-1	0.1700	0.1648	0.1663
377-2	0.1681	0.1653	0.1684
377-3	0.1699	0.1747	0.1641
377-4	0.1665	0.1645	0.1661
377-5	0.1709	0.1674	0.1682
377-6	0.1696	0.1738	0.1664
377-7	0.1690	0.1641	0.1675
377-8	0.1720	0.1719	0.1662
382-1	0.1531	0.1479	0.1514
382-2	0.1522	0.1566	0.1444
382-3	0.1509	0.1523	0.1441
382-4	0.1543	0.1462	0.1575
382-5	0.1537	0.1538	0.1543
382-6	0.1529	0.1532	0.1461
382-7	0.1493	0.1381	0.1487
382-8	0.1507	0.1481	0.1437
383-1	0.1518	0.1499	0.1449
383-2	0.1512	0.1556	0.1448
383-3	0.1510	0.1436	0.1516
383-4	0.1512	0.1486	0.1545
383-5	0.1506	0.1458	0.1541
383-6	0.1526	0.1472	0.1524
383-7	0.1493	0.1450	0.1478
383-8	0.1510	0.1492	0.1480

NOTE: Measurements were taken on the widthwise center of the specimens at spanwise center and at 0.250 inch from each end.

TABLE II

SCREENING TEST SPECIMEN DIMENSIONS

Specimen Number	Thickness (Inch)		
	Center	End #1	End #2
1C-1	0.1683	0.1664	0.1685
1C-2	0.1680	0.1668	0.1673
1C-3	0.1666	0.1680	0.1683
1C-4	0.1679	0.1668	0.1681
1C-5	0.1681	0.1677	0.1685
2C-1	0.1380	0.1361	0.1380
2C-2	0.1373	0.1355	0.1373
2C-3	0.1353	0.1370	0.1379
2C-4	0.1372	0.1380	0.1385
2C-5	0.1354	0.1345	0.1357
2C-6	0.1358	0.1346	0.1353
3C-1	0.1193	0.1159	0.1198
3C-2	0.1183	0.1192	0.1197
3C-3	0.1200	0.1174	0.1196
3C-4	0.1186	0.1183	0.1191
3C-5	0.1196	0.1155	0.1177
3C-6	0.1200	0.1187	0.1188

NOTE: Measurements were taken on the widthwise center of the specimens at spanwise center and at 0.250 inch from each end.

TABLE III

FOUR-POINT BEND SCREENING TEST RESULTS

Specimen No.	Test Temp (°C)	Test Mode	Test Rate	Maximum Fatigue Load (lb)	Minimum Fatigue Load (lb)	Cycles to Failure	Failure Modes and Locations
377-1	400	Tens. Fracture	0.050/min	53	0	N/A	2 Places in center span
377-2	400	Compr. Fracture	0.050/min	801	0	N/A	Under right load pin; specimen end #1
377-3	400	Compr. Fatigue	10 Hz	N/A	N/A	1/4	Under inner load pins
377-4	400	Compr. Fatigue	10 Hz	480	140	740	Under left load pin; specimen end #2
377-5	RT	Compr. Fatigue	5 Hz	542	32	4,670	Under right load pin; specimen end #1
377-6	RT	Compr. Fatigue	5 Hz	540	28	6,190	Under right load pin; specimen end #2
377-7	RT	Compr. Fatigue	5 Hz	452	34	110	Under left load pin; specimen end #1
377-8	--	Not Tested	--	--	--	--	--
382-1	400	Compr. Fracture	0.050/min	868	0	N/A	Under right load pin; specimen end #1
382-2	400	Compr. Fracture	0.050/min	896	0	N/A	2 places in center span, under left load pin, 1 place in left load span; specimen end #2
382-3	400	Compr. Fatigue	5 Hz	443	20	2,740	2 places in center span; 1 place in right load span; specimen end #1
382-4	400	Compr. Fatigue	5 Hz	410	32	38,050	Substrate failure in center span
382-5	RT	Compr. Fatigue	5 Hz	426	22	211,080	Substrate failure in center span
382-6	RT	Compr. Fatigue	5 Hz	424	32	156,050	Substrate failure under right load pin; specimen end #2
382-7	RT	Compr. Fatigue	5 Hz	425	28	141,840	Substrate failure under left load pin; specimen end #2
382-8	RT	Compr. Fatigue	5 Hz	428	32	105,300	Not a substrate failure; 1 place in center span
383-1	400	Compr. Fracture	0.050/min	416	0	N/A	1 place in left load span; specimen end #2
383-2	400	Compr. Fracture	0.050/min	557/670	0	N/A	Under right load pin; specimen end #2
383-3	RT	Compr. Fatigue	5 Hz	426	32	710	Under right load pin; specimen end #1 (twisted specimen)
383-4	RT	Compr. Fatigue	5 Hz	420	28	21,240	Under both inner load pins
383-5	RT	Compr. Fatigue	5 Hz	422	25	16,060	Under right load pin; specimen end #1
383-6	--	Not Tested	--	--	--	--	--
383-7	RT	Compr. Fatigue	5 Hz	424	23	4,530	Under right load pin; specimen end #1
383-8	--	Not Tested	--	--	--	--	--

TABLE IV

FOUR-POINT BEND SCREENING TEST RESULTS

Specimen No.	Test Temp (°C)	Test Mode	Test Rate	Maximum Fatigue Load (lb)	Minimum Fatigue Load (lb)	Cycles to Failure	Failure Modes and Locations
1C-1	RT	Compr. Fracture	0.050/min.	450	0	N/A	Outside of left load pin; specimen end #2
1C-2	RT	Compr. Fracture	0.050/min.	438	0	N/A	Outside of left load pin; specimen end #1
1C-3	--	Not Tested	--	--	--	--	--
1C-4	--	Not Tested	--	--	--	--	--
1C-5	--	Not Tested	--	--	--	--	--
2C-1	RT	Compr. Fatigue	5 Hz	343	29	40	Multiple breaks in coating within center span
2C-2	RT	Compr. Fatigue	5 Hz	348	25	910	Under left load pin; specimen end #2
2C-3	RT	Compr. Fatigue	5 Hz	345	34	2,890	2 places in center span, outside of right load pin; specimen end #1
2C-4	RT	Compr. Fatigue	5 Hz	347	25	210	1 place in center span, under right load pin; Specimen end #1
2C-5	RT	Compr. Fracture	0.050/min.	402	0	N/A	Under right load pin; specimen end #1
2C-6	RT	Compr. Fracture	0.050/min.	430	0	N/A	Under left load pin; specimen end #2
3C-1	RT	Compr. Fatigue	5 Hz	267	18	23,420	Under left load pin; specimen end #2
3C-2	RT	Compr. Fatigue	5 Hz	269	20	159,120	Under left load pin;* specimen end #2
3C-3	RT	Compr. Fatigue	5 Hz	267	16	185,710	Inside left load pin; specimen end #2
3C-4	RT	Compr. Fatigue	5 Hz	270	19	80,350	Under both load pins
3C-5	RT	Compr. Fracture	0.050/min.	452	0	N/A	Inside right load pin; specimen end #1
3C-6	RT	Compr. Fracture	0.050/min.	454	0	N/A	Inside left load pin; specimen end #2

*There were signs of some erosion under the load pin.

TABLE V

DATA BASE FATIGUE SPECIMENS

Specimen Number	Thickness (Inch)			Test Mode	Test Temp (°C)	Test Rate	Maximum Fatigue Load (lb)	Minimum Fatigue Load (lb)	Cycles to Failure	Comments on Failures
	Center	End #1	End #2							
426-Ti-42	0.1694	0.1777	0.1824	Compr. Fracture	RT	0.050/min	583.00	0	N/A	Under R-LP
426-Ti-43	0.1811	0.1833	0.1835	Compr. Fracture	RT	0.050/min	586.00	0	N/A	Under L-LP
426-Ti-44	0.1745	0.1837	0.1850	Compr. Fatigue	RT	5Hz	356.00	24	94,990	Multiple failure
426-Ti-45	0.1688	0.1754	0.1773	Compr. Fatigue	RT	5Hz	354.00	25	55,800	Substrate failure
426-Ti-47	0.1594	0.1677	0.1726	Compr. Fatigue	RT	5Hz	250.00	18	192,550	Substrate failure
426-Ti-48	0.1631	0.1766	0.1813	Compr. Fatigue	RT	5Hz	250.00	18	188,600	Substrate failure
426-Ti-49	0.1651	0.1724	0.1755	Compr. Fatigue	RT	5Hz	250.00	19	10,000,000	Runout
426-Ti-54	0.1675	0.1743	0.1776	Compr. Fatigue	RT	5Hz	250.00	17	1,308,700	Substrate failure, ceramic @ L-LP
426-Ti-55	0.1673	0.1751	0.1763	Compr. Fatigue	RT	15Hz	300.00	21	148,600	Ceramic @ R-LP, delaminated
426-Ti-56	0.1695	0.1755	0.1769	Compr. Fatigue	RT	15Hz	275.00	20	4,904,800	Substrate failure, ceramic @ R-LP
426-Ti-57	0.1551	0.1660	0.1663	Compr. Fatigue	RT	15Hz	398.00	220	1,076	Ceramic @ R-LP
426-Ti-58	0.1652	0.1741	0.1783	Compr. Fatigue	RT	15Hz	350.00	210	8,724,000	Delamination outside R-LP
426-Ti-59	NA	NA	NA	NA	--	--	--	--	--	Specimen not tested
426-Ti-60	0.1570	0.1662	0.1692	Compr. Fatigue	RT	15Hz	350.00	210	430,000+	Failure in outside span & @L-LP; radiused delam.
426-Ti-61	0.1695	0.1799	0.1823	Compr. Fatigue	RT	15Hz	300.00	19	2,894,900	Ceramic @ R-LP & multiple in midspan
426-Ti-62	0.1700	0.1787	0.1802	Compr. Fatigue	RT	1-5Hz	420.00	29	5,340	Ceramic @ L-LP

NOTE: Specimen substrates, starting with 426 Ti-49, were polished!
R-LP = right load pin
L-LP = left load pin

TABLE V (CONCLUDED)

DATA BASE FATIGUE SPECIMENS

Specimen Number	Thickness (Inch) Center	End #1	End #2	Test Mode	Test Temp (°C)	Test Rate	Max. Fatigue Load (lb)	Min. Fatigue Load (lb)	Cycles to Failure	Comments on Failures
426-17-21	0.1470	0.1451	0.1531	Compr. Fracture	RT	0.050/min	400.0	0	N/A	--
426-17-22	0.1454	0.1383	0.1468	Compr. Fracture	RT	0.050/min	378.0	0	N/A	--
426-17-23	NA	NA	NA	Not Tested	--	--	--	--	--	Ceramic damage during polishing
426-17-25	0.1465	0.1471	0.1493	Compr. Fatigue	RT	15Hz	295.0	20	1,400	Ceramic @ R-LP
426-17-26	0.1485	0.1501	0.1510	Compr. Fatigue	RT	15Hz	300.0	20	1,640	Ceramic @ L-LP
426-17-27	0.1445	0.1430	0.1461	Compr. Fatigue	RT	5Hz	298.0	21	2,378	Ceramic @ L-LP
426-17-28	0.1473	0.1491	0.1510	Compr. Fatigue	RT	15Hz	250.0	18	83,170	Ceramic @ R-LP & delamination
426-17-29	0.1359	0.1400	0.1453	Compr. Fatigue	RT	15Hz	210.0	19	1,235,000	Ceramic @ R-LP & in mid-span
426-17-30	0.1460	0.1485	0.1500	Compr. Fatigue	RT	15Hz	225.0	17	1,504,700	Ceramic @ R-LP & in mid-span; (O.L. @ rem)
426-17-31	0.1459	0.1482	0.1508	Compr. Fatigue	RT	15Hz	425.0	29	823	Ceramic @ L-LP & mid-span/aluminum pads
426-17-32	0.1385	0.1438	0.1412	Compr. Fracture	RT	0.050/min	547.8	0	N/A	Ceramic @ R-LP/aluminum pads
426-17-34	0.1521	0.1523	0.1542	Compr. Fracture	RT	0.050/min	662.1	0	N/A	Ceramic @ LP/copper pads (37.5 mm R)
426-17-35	0.1425	0.1466	0.1487	Compr. Fatigue	RT	15Hz	432.0	30	480	Ceramic @ R-LP & center/copper pads (37.5 mm R)
426-17-36	NA	NA	NA	NA	RT	--	--	--	--	Specimen not tested
426-17-37	NA	NA	NA	NA	RT	--	--	--	--	Specimen not tested
426-17-38	NA	NA	NA	NA	RT	--	--	--	--	Specimen not tested
426-17-39	0.1435	0.1397	0.1402	Compr. Fracture	RT	0.050/min	700.8	0	N/A	Ceramic @ R-LP/copper pads (150 mm R)/wide contacts

NOTE: Specimen substrates, starting with 426 Ti-49, were polished!
R-LP = right load pin
L-LP = left load pin

TABLE VI

DATA BASE FATIGUE SPECIMENS

Specimen Number	Thickness (Inch)			Test Mode	Test Temp (°C)	Test Rate	Maximum Fatigue Load (lb)	Minimum Fatigue Load (lb)	Cycles to Failure	Comments on Failures
	Center	End #1	End #2							
426-B-1	0.0816	0.0780	0.0848	Tens. Fracture	RT	0.050/min.	10.30	0	N/A	–
426-B-2	0.0866	0.0865	0.0867	Tens. Fracture	RT	0.050/min.	11.16	0	N/A	–
426-B-3	0.0882	0.0878	0.0893	Tens. Fatigue	RT	10Hz	9.00	0.64	1,220	Failure @ L-LP
426-B-4	0.0848	0.0857	0.0860	Tens. Fatigue	RT	10Hz	7.00	0.49	83,450	Failure @ R-LP
426-B-5	0.0887	0.0888	0.0894	Tens. Fatigue	RT	10Hz	6.00	0.42	3,441,000	Failure @ R-LP
426-B-6	0.0889	0.0885	0.0894	Tens. Fatigue	RT	10Hz	8.00	0.56	35,820	Failure @ approximate center
426-B-7	0.0862	0.0862	0.0871	Tens. Fracture	400	0.050/min.	19.30	0	N/A	Failure @ R-LP
426-B-8	0.0879	0.0861	0.0885	Compr. Fracture	400	0.050/min.	19.70	0	N/A	Failure @ L-LP
426-B-9	0.0841	0.0856	0.0878	Tens. Fracture	RT	0.050/min.	17.00	0	N/A	30 min. soak @ 400F; test @ RT; midspan failure
426-B-10	0.0885	0.0885	0.8860	Tens. Fatigue	400	10Hz	15.50	1.09	No data	–
426-B-11	0.0870	0.0882	0.0887	Tens. Fatigue	400	10Hz	13.50	0.95	6,672,200	–
426-B-12	0.0848	0.0861	0.0866	Tens. Fatigue	400	10Hz	15.00	1.05	1,276	Failure @ L-LP
426-B-13	0.0897	0.0896	0.0897	Tens. Fatigue	400	10Hz	15.60	1.09	2,153	–
426-B-14	0.0882	0.0893	0.0896	Tens. Fatigue	400	10Hz	14.20	0.99	10,000,000	Runout - no failure
426-B-15	0.0887	0.0877	0.0888	Tens. Fracture	400	0.050/min.	19.20	0	N/A	Aged: 40 hrs @ 770F;
426-B-16	0.0852	0.0865	0.0870	Tens. Fatigue	400	2Hz	15.14	1.06	135,050	Aged: 40 hrs @ 770F; failure under load point
426-B-17	0.0885	0.0881	0.0891	Tens. Fatigue	400	2 & 10 Hz	16.25	1.14	9,420,000	Aged: 40 hrs @ 770F; failure under load point
426-B-18	0.0884	0.0884	0.0886	Tens. Fatigue	RT	1Hz	16.00	1.2	79	Aged: 40 hrs @ 770F; failure under load point
426-B-19	0.0825	0.0819	0.0843	Tens. Fatigue	RT	2 to 10Hz	12.50	0.9	188,590	Aged: 40 hrs @ 770F; failure under load point;
426-B-20	0.0835	0.0844	0.0852	Tens. Fatigue	RT	2 to 10Hz	12.00	0.84	No data	Machine did not shut off after specimen failure; apparent multiple loading points on specimen surface.

NOTE: R-LP = right load pin
L-LP = left load pin

TABLE VII

DATA BASE FATIGUE SPECIMENS

Specimen Number	Thickness (Inch)			Test Mode	Test Temp (°C)	Test Rate	Maximum Fatigue Load (lb)	Minimum Fatigue Load (lb)	Cycles to Failure	Comments on Failures
	Center	End #1	End #2							
360-17-5	0.1375	0.1388	0.1390	Compr. Fracture	RT	0.050/min.	939.6	0	N/A	"Fish-Scale" type failure approx. 1/8 in. from R-LP in midspan
360-17-6	0.1286	0.1362	0.1401	Compr. Fatigue	RT	5 Hz	650.0	45.5	42,439	Copper pads; friction & wear type failure at pads and substrate under load points
360-17-7	0.1250	0.1352	0.1407	Compr. Fatigue	RT	5 Hz	614.0	42.0	493	Failure at midspan/spallation type
360-17-8	0.1334	0.1336	0.1340	Compr. Fatigue	RT	5 Hz	550.0	39.0	2,030	Failure at midspan/spallation type
3C-38	0.1269	0.1273	0.1279	Compr. Fracture	RT	0.050/min.	888.0	0	N/A	"Peel" type failure in midspan
3C-39	--	--	--	--	--	--	--	--	--	Specimen not tested
3C-40	--	--	--	--	--	--	--	--	--	Specimen not tested

NOTE: R-LP = right load pin

TABLE VIII

DATA BASE FATIGUE TEST RESULTS

Specimen Number	Thickness (Inch)			Test Mode	Test Temp (°C)	Test Rate	Maximum Fatigue Load (lb)	Minimum Fatigue Load (lb)	Cycles to Failure	Comments on Failures
	Center	End #1	End #2							
25	0.1233	–	–	Compr. Fracture	RT	0.050/min.	760	0	NA	–
26	0.1247	–	–	Compr. Fracture	RT	0.050/min.	734	0	NA	–
27	0.1240	–	–	Compr. Fatigue	RT	1-5 Hz	508	35.6	17,190	Blisters prior to failure; "good" failure
28	0.1247	–	–	Compr. Fatigue	RT	1-5 Hz	580	40.6	35,000	Blisters @ 20,000; "good" failure
32	0.1244	–	–	Compr. Fatigue	RT	5 Hz	522	36.5	33,190	Substrate failure/multiple initiation
33	0.1225	0.1240	0.1234	Compr. Fatigue	RT	5 HZ	419	29.3	98,380	Substrate failure
34	0.1234	0.1248	0.1248	Compr. Fatigue	RT	5 HZ	403	28.2	120,240	Substrate failure
36	0.0762	0.0777	0.0780	Compr. Fatigue	RT	2 Hz	130	9.1	164,640	Ceramic failure at load point
38	0.0785	0.0788	0.0789	Compr. Fatigue	RT	2 Hz	139	9.7	158,060	Substrate failure

4 Conclusions and Recommendations

The testing performed was not totally satisfactory. The four-point bend test set-up which is so common in ceramics testing has been found unacceptable as a means for defining the fatigue strength of the material for high cycle conditions. The failure mode of surface layer spallation, observed in the engine testing program, was demonstrated to be a real failure mode in compression tests, but was difficult to achieve in the four-point bend testing.

Stress conditions in four-point bend testing are quite nonuniform. The contact stresses are substantial at the surface and for the layers of material and interfaces below the contact point. Curvature effects in bending as well as tolerance effects between the specimen and the loading bars can cause additional local stress conditions which can induce premature static and fatigue failures.

Long life fatigue testing is particularly challenging for specimens with nonuniform loading and internal stress conditions. Failure modes include specimen modes as well as material modes. The test program found that the surface roughness and, possible, residual stress conditions on the metallic substrate were extremely sensitive parameters at the desired test conditions. Polishing the substrate had only limited success in removing premature, specimen configuration fatigue modes.

Stress gradients, in four-point bend testing, result in material response that is only strictly accurate for the bend test itself. Uniform stresses in a well-defined gage section are highly desirable conditions for strength and fatigue testing. This is particularly true for ceramic materials where defect sizes play a strong role in both static and fatigue strength models. The four-point bend test results can be expected to be dependent on the thickness of the ceramic and the size of the specimen. The test matrix did not permit this evaluation to be confirmed.

Unfortunately, the reason that the four-point bend specimen is widely used for ceramics testing is that the test arrangement is relatively inexpensive. Additionally, other test specimen configurations for ceramics have not been developed that fully address the technical limitations of the four-point bend test.

Some limited thought was given to other, more suitable test arrangements for ceramic coatings. Monolithic ceramic test specimens of a reasonable size are virtually impossible to fabricate without the use of a metallic substrate. However, coating of a substrate and then removing the substrate through chemical etching is a possible means of test specimen fabrication. Pure compressive testing of a uniform tube of ceramic poses the difficulty of achieving good load introduction at the specimen ends in order to avoid local fracture, as well as the influence of friction constraint at the platten-specimen interface.

Tension testing of ceramics is even more difficult due to stress concentrations in the regions of specimen grips or the transitions to the gage section, or both. The use of the "Brazil" specimen for tension testing has been shown successful in earlier ceramic coating testing at SwRI. The specimen has the shape and proportions of a "Tums" tablet and is loaded in compression. The specimen internal stress distribution is a nearly uniform tensile stress across the plane between the load contact points. Tensile static and fatigue strength results have been successfully achieved for this specimen.

Rotating beam, ceramic coated tubular specimens were considered for high-cycle fatigue testing. The rotating beam test also has some substantial drawbacks. One of the principal test condition requirements for the current program was for high, steady stress together with superimposed cyclic stresses. The rotating beam test arrangement would need to include a steady, prestress in the coated tube; no such standard rotating-beam test machine is available. Design and fabrication of such a test machine is possible, but is judged to be relatively expensive, particularly when considering the high temperature requirements. Additionally, the load transition from the substrate into the ceramic coating in the test section is likely to produce its own, unique fatigue failures.

In conclusion, therefore, the testing did achieve partial success in spite of the inherent problems of the four-point bend specimen configuration. The use of simpler, uniform-stress test specimens is recommended for material evaluation. Tubular compression specimens and the "Brazil" specimen for tension testing are recommended as alternatives to the bending specimen.