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FINAL REPORT

ON

DURIP95/ULTRA HIGH PRECISION DIAGNOSTIC HIGH TEMPERATURE LABORATORY

(AFOSR GRANT # F49620-95-1-0483)

to

Air Force Office of Scientific Research Bolling AFB Washington D.C.

from

Professor Golam M. Newaz Principal Investigator Mechanical Engineering Department Wayne State University Detroit, MI 48202

July 18, 1997

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PROJECT SUMMARY

The DURIP grant was used to develop an efficient high temperature laboratory with high precision instruments to make deformation and load measurements in high temperature materials including advanced composites. The laboratory has been successfully developed in Mechanical Engineering Department at Wayne State University under the direction of Professor Golam Newaz - who was the PI of the program. Based on the capability developed, the laboratory can be considered a national resource as extensive capabilities have been integrated with existing facilities in Advanced Composites Research Laboratory at Wayne State University. High temperature testing capabilities include evaluation of mechanical and thermomechanical capabilities up to 2200 F with an induction heating system that is interfaced with modern MTS 810 servohydraulic equipment. Polymeric, ceramic and metal matrix composites including superalloys with thermal barrier coatings can be tested for material properties, fatigue and fracture performance. For aerospace engine applications, a wide variety of material systems with high temperature performance can be evaluated in our laboratory. Another critical competency achieved was in the area of post-test evaluation. An excellent microscopy facility has been developed that can be utilized to interrogate material damage development, progressive cracking, any plastic deformation, material defects and failure modes. An optical microscope with all necessary polishing facilities and ptotomicrography is now available to investigate deformation and failure in high temperature materials. We are now in the implementation stage to equip our laboratory with a Hopkinson Pressure Bar with high temperature capabilities. With the combined high temperature testing capabilities, the research and teaching environment at WSU shows great promise with regard to advanced high temperature material testing capabilities. With our capabilities we have attracted research dollars from other Air Force

programs through Small Business organizations to the tune of \$300K. There is now additional requests to funding agencies of about \$700K that utilizes the capability of the laboratory. A Ph. D. was granted and there are three Ph.D. students currently utilizing the equipment. A strong educational base has been created as well due the development of this laboratory. The DURIP95 award was for \$275,000. We have expended a total of \$274,261.02 for the program.

The overall high temperature testing capabilities in Mechanical Engineering Department at Wayne State University adds a new strength in educational and research capability of the department. Professor Newaz's expertise is in the area of damage evolution assessment, mechanism-based constitutive response modeling, fatigue and fracture evaluation with emphasis on experimental techniques. The DURIP95 grant was utilized to develop a combined educational and research capability that will continue to help the PI for a long time to come in his research career. Instruments purchased under the DURIP program are listed in the purchase orders enclosed.

A recent professional resume of the PI is attached to show the impact of this laboratory on research accomplishments of the PI including publications. The laboratory is considered vital and without the facilities, attracting research dollars would not be possible. A number of representative publications are also attached which were a direct result of the availability of the instruments in the laboratory.

The laboratory was instrumental for Dr. Saied Nusier to obtain his Ph.D. in February, 1997. Current graduate students include, Mr. M. Abdel-Haq and Mr. Ke Zhang who are utilizing the high temperature laboratory for their thesis work.

LIST OF INSTRUMENTS ORDERED

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I	# U–R255	Brightfield/Darkfield Quintuple Nosepiece	\$ 578.16	\$ 425.92 \$ 578.16
1	# 4- U 113	Rectangular Mechanical Stage for Olympus B-MAX microscopes	\$ 401.28	\$ 401.28
1	#6-U110	Abbe Condenser	\$ 122.32	\$ 122 32
1	# U − M501	Brightfield Beamsplitter Cube	\$ 134.64	\$ 134 64
1	#U– M503	Darkfield Beamsplitter Cube:	\$ 165.44	\$ 165 //
1	#1-UR522	Universal Plan Fluorite 5x Transmitted Brightfield	\$ 727.00	\$ 727.00
1	# 1–UR523	Universal Plan Fluorite 10x Transmitted Brightfield	\$ 999.00	\$ 999.00 (m)
1	# 1–UR525	Universal Plan Fluorite 20x Transmitted Brightfield	\$1185.00	\$1185.00
1	#1−UR530	Universal Plan Fluorite 50x Transmitted Brightfield	\$1486.00	\$1486.00
1	#1-UR534	Universal Plan Fluorite 100x Transmitted Brightfield	\$2287.00	\$2287.00
1	#2– U504	10x helical superwidefield eyepiece	\$ 290.40	\$ 290.40
1	# U−P210	Polarizer Slider	\$ 439.12	\$ 439.12
1	#U-P219	Analyzer for polarized light	\$ 613.36	\$ 613.36
1	#U P110	Polarizer for Transmitted light	\$ 154.88	\$ 154.88
1	#9− U573	Filter Slider	\$ 52.80	\$ 52.80
1	#9 −0571	Filter Slider	\$ 52.80	\$ 52,80
1	#9 U579	Filter Slider	\$ 18.48	\$ 18.48
1	# U−P200	DIC Prism Slider	\$2492.16	\$2492.16
1	#9 –U520	IF550 Filter Slider	\$ 72.16	\$ 72.16
1	# 9−B323	45mm diameter Interference Green Filter	\$ 102.96	\$ 102.96
1	# UP212	POLARIZER TINT PLATE (530NM)	\$ 791.12	\$ 791.12
1	#9 -0510	Filter Slider	\$ 45.76	\$ 45.76
1	# 7–U311	AUTO EXPOSURE CONTROL BOX	\$3937.12	\$3937.12

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Purchase Requisition # G011635 (con't) Pg. 2

Quantity	Part No.	& Description	<u>Unit Cost</u>	<u>Total</u>
1	#7– ∪301	AUTO EXPOSURE BODY-PM30	\$2544.08	\$2544.08 (
1	#2− U730	3.3X Photoeyepiece	\$ 227.92	\$ 227.92
1	# 7−U510	Camera Adapter	\$ 195.36	\$ 195.36
1	#7 − 0 530	35mm Camera Back for DX Coded Film	\$ 752.40	\$ 752.40/
1	#7- U620	Adapter for front mounting of Large Format Camera Backs.	\$ 938.96	\$ 938.96
1	# 7–₩640	Intermediate Adapter for 4" x 5" Large Format Film Holder.	\$ 511.28	\$ 511.28
1	#617878	Polarid "545" sheet film holder	\$ 234.08	\$ 234.08
1	# 7–∪840	Bright Frame Focusing Viewfinder	\$ 510.40	\$ 510.40
1	#D63BXC	Diagnostic Instruments 0.63X C-Mount	\$ 263.12	\$ 263.12

*Olympus Microscopes and accessories are warranted from date of purchase for five (5) years of mechanical and optical components and one (1) year on electrical and electronic components. The Olympus AX70, IX70, and IX50 Microscopes and Microtomes are warranted for one (1) year from the date of purchase on all components. All other accessories, parts or equipment not manufactured by LECO are covered to the extent of warranty provided by the original manufacturer.

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WAYNE STATE UNIVERSITY

PROFESSIONAL RECORD

Name: Office Address Telephone No:	Golam Newaz 2135 Mechanical Eng (313) 577-3877	gineering
DEPARTMEN	T: Mechanical Engineer	ring
PRESENT RAI	NK & DATE OF RANK:	Professor (January 1, 1995)
CITIZEN OF:		United States of America
EDUCATION:		
High School: Baccalaureate: Graduate:	Dhaka College, Dhaka, Bang Nuclear Engineering, Texas M.STheoretical & Applied Champaign	ladesh A&M University Mechanics, University of Illinois @ Urbana-
Ph.D.:	Theoretical & Applied Mech	anics, University of Illinois @ Urbana-Champaign
Postgraduate: Licensure: Certification:	N/A N/A N/A	

FACULTY APPOINTMENTS AT OTHER INSTITUTIONS: N/A

PROFESSIONAL SOCIETY MEMBERSHIP(S):

Member, American Society of Mechanical Engineers, 1987-present Member, American Society of Testing and Materials, 1982-present Member, American Society for Composites, 1993-present Member, U.S. Japan Council on Composites, 1992-present

HONORS/AWARDS: Best Paper Award, Society of Plastics Industry Annual Technical Conference, 1983.

BIOGRAPHICAL CITATIONS: Not Checked

I. TEACHING

- A. Years at Wayne State:
 - 1. ME 772 Advanced Mechanics of Composites, Winter 1995, Winter 1996, Winter 1997.
 - 2. ME 795 Delamination in Advanced Composites-Directed Study, Winter 1995

- 3. ME 572 Mechanics of Composite Materials, Fall 1995
- 4. ME 562 Fracture Mechanics in Engineering Design, Fall 1996
- B. Years at Other Colleges/Universities:
 - University of Illinois @ Urbana-Champaign TAM 110 Engineering Statics, Fall 1977 TAM 110 Engineering Statics, Winter 1978 TAM 210 Engineering Dynamics, Fall 1978 TAM 110 Engineering Statics, Winter 1979 TAM 110 Engineering Statics, Fall 1979
- C. Courses Taught at Wayne State in Last Five Years:
 - 1. Undergraduate: BE 206 Problem Solving & Design
 - Graduate: ME 772 Advanced Mechanics of Composites, Winter 1995 ME 795 Delamination in Advanced Composites (Directed Study), Winter 1995. ME 572 Mechanics of Composite Materials, Fall 1995 ME 562 Fracture Mechanics in Engineering Design, Fall 1996.
 - 3. Graduate Professional School: N/A
- D. Essays/Theses/Dissertations Directed:

M.S. Theses:

- 1. "Analysis of Mixed-Mode Crack Growth in CMC", M. Krishnappa, July 1996.
- 2. "Evaluation of Innovative Strain-Gage Application to Polymer Test Samples", S. Sterbenz, October 1996.

Ph.D. Dissertations:

1. "Damage Accumulation and Failure in TBC", S.Q. Nusier, Feb. 1997.

II. <u>RESEARCH</u>

- A. Research in Progress:
 - 1. Inelastic Deformation Mechanisms in Metal Matrix Composites in Compression
 - 2. Damage Accumulation Mechanisms in Thermal Barrier Coated Superalloys
 - 3. Analysis of Mixed-Mode Crack Growth in Ceramic Matrix Composite
 - 4. Crushing & Energy Absorption in Composite Tubes
 - 5. Tribological Behavior of Cylinder Liner Materials
- B. Funded Research in Last Five Years

- 1. "Inelastic Deformation Mechanisms in Metal Matrix Composites in Compression", G. Newaz-Principal Investigator, AFOSR, \$319,000 over two years, Feb. 1993-Dec. 1994.
- 2. "Life Prediction of Ceramic Matrix Composite", G. Newaz-Principal Investigator, Air Force Materials Directorate, PRDA IV Program with Pratt & Whitney and UTRC, \$550,000 over three years, Oct. 1993-present.
- "Thermomechanical Fatigue of Metal Matrix Composites", G. Newaz and B. Majumdar Co-Principal Investigators, NASA NRA Program, \$154,000 over two years, January 1993-December 1994.
- "Isothermal Fatigue Mechanisms in Metal Matrix Composite", G. Newaz and B. Majumdar Co-Principal Investigators, NASA NRA Program, \$140,000 over one year, January 1992-December 1992.
- 5. "Inelastic Deformation Mechanisms under Monotonic Loading in Metal Matrix Composites", G. Newaz and B. Majumdar Co-Principal Investigators, NASA NRA Program, \$125,000 over one year, January 1991-December 1991.
- 6. "Delamination Growth in Ceramic Matrix Composites", G. Newaz-Principal Investigator, GE/NASA EPM Program, \$84,000 over 1.5 years, January 1994present.
- 7. "Damage Accumulation Mechanisms in Thermal Barrier Coatings", G. Newaz-Principal Investigator, AFOSR, \$240,000 over three years, March 1995present.
- 8. "Life Prediction of Ceramic Composites", G. Newaz-Principal Investigator, Battelle/U.S. Air Force, \$39,500, August 1995-December 1996.
- 9. "High Temperature Laboratory Development", G. Newaz-Principal Investigator, AFOSR DURIP Grant, \$275,000, June 1995-February 1997.
- 10. "Design Tool for Oxidation in TBC", G. Newaz-Principal Investigator, RAI/U.S. Army, \$24,500, January 1996-December 1996.
- 11. "Dual Applications of MMC: Phase I", G. Newaz-Principal Investigator, RAI/U.S. Air Force, \$21,000, June 1995-May 1996.
- 12. "Damage Tolerance of PMC", G. Newaz-Principal Investigator, \$21,000, RAI/U.S. Air Force, January 1996-December 1996.
- "Dual Applications for MMC: Phase II", G. Newaz-Principal Investigator, \$224,000, RAI/U.S. Air Force, June 1996-May 1998.
- 14. "REU Program-Summer Internship", Co-PI-G. Newaz and R.F. Gibson, Ford Motor Company, \$20,000, 1996.
- 15. "REU Program-Summer Internship", Co-PI-G. Newaz and R.F. Gibson, Ford Motor Company, \$16,500, 1997.
- 16. "Advances in Composites Manufacturing Workshop", Co-PI G.Newaz and R.F. Gibson, National Science Foundation, \$19,161, 1997.

 17. "Fatigue Methodology for Short-Fiber Composites", G. Newaz - Principal Investigator, Automotive Composites Consortium (Ford/GM/Chrysler), \$33,900, 1997

III. <u>PUBLICATION</u>

- A. Scholarly Books Published
 - 1. Newaz, G.M., (with Sierakowski, R.L.), "Damage Tolerance of Advanced Composites", Technomic Publishing Co., Lancaster, PA, 1995.
- B. Chapters Published
 - 1. Newaz, G.M., "Polymer Composites", in Materials Science and Technology Series, VCH, Weinham, Germany, 1994.
- 2. Newaz, G.M., "Mechanical Behavior of Metal Matrix Composites", in Advances in Metal Matrix Composites, in press, S. Mall and T. Nicholas, Editors, Technomic Publication Co., Lancaster, PA, 1997.
- C. Editorships of Book/Proceedings
 - 1. Newaz, G.M., "Delamination of Advanced Composites", Technomic Publishing Co., Lancaster, PA, 1993.
- D. Journal Articles Published

Refereed Journals

- 1. Newaz, G.M., Nusier, S.Q., and Chaudhury, Z.A., 1996, "Damage Accumulation Mechanisms in Thermal Barrier Coatings", accepted for publication in <u>ASME J. of Engineering Materials and Technology</u>, 1997.
- 2. Chaudhury, Z.A., Newaz, G.M., Nusier, S.Q., and Ahmed, T., "Interfacial Damage in EB-PVD Thermal Barrier Coatings Due to Thermal Cycling", in press, J. of Materials Science and Engineering, 1996.
- 3. Nusier, S.Q., and Newaz, G.M., "Analysis of Interfacial Cracks in a TBC/Superalloy System Under Thermomechanical Loading", accepted for publication in the <u>ASME J. of Engineering for Gas Turbines and Power</u>, 1997.
- 4. Newaz, G.M., and Bonora, N., "Fatigue Life Modeling of Hybrid CMC", <u>ASTM STP 1309, Amer. Soc. for Testing and Materials</u>, 1996.
- 5. Rahman, M. and Newaz, G.M., "Elastostatic Surface Displacements of a Half-Space Reinforced by a Thin Film Due to an Axial Ring Load", in press, International Journal of Engineering Science, 1996.
- 6. Bonora, N., and Newaz, G.M., "Modeling Damage Evolution in a Hybrid Ceramic Matrix Composite Under Static Tensile Load", in press, <u>ASME J. of</u> <u>Engineering Materials and Technology</u>, 1997.

- 7. Bonora, N., and Newaz, G.M., "Low-Cycle Fatigue Life Estimation for Ductile Metals Using a Nonlinear Continuum Damage Mechanics Model", accepted for publication in <u>International J. of Solids and Structures</u>, 1997.
- Newaz, G.M., and Majumdar, B.S., "In-Phase Thermomechanical Fatigue of MMC Deformation Response and Progressive Damage Mechanisms", <u>ASTM</u> <u>STP 1274</u>, 264, (1996).
- 9. Newaz, G.M., "Constitutive Response and Deformation Mechanisms in Unidirectional MMC Under Compression", <u>ASTM STP 1274</u>, 278, (1996).
- Newaz, G.M., Majumdar, B.S., Brust, F.W., "Inelastic Deformation Mechanisms in SCS-6/Ti 15-3 MMC Lamina Under Compression", <u>ASTM</u> <u>STP 1258</u>, (1996).
- Newaz, G.M., and Majumdar, B.S., "Failure Modes in Transverse MMC Lamina Under Compression", <u>J. of Materials Science & Letters</u>, UK, 12: 551-552, (1993).
- Newaz, G.M., and Majumdar, B.S., "A Comparison of Mechanical Response of MMC at Room and Elevated Temperatures", <u>J. of Composites, Science &</u> <u>Technology</u>, UK, 50: 85-90, (1993).
- Newaz, G.M., Majumdar, B.S. and Brust, F.W., "Thermal Cycling Response of Quasi-Isotropic Metal-Matrix Composite", <u>J. of Eng. Materials and</u> <u>Technology</u>, Vol. 114, #2, 156-161, (1992).
- 14. Newaz, G.M., Majumdar, B.S., "Fatigue Crack Initiation Around Holes in MMC", <u>Engineering Fracture Mechanics</u>, Vol. 42, #4, 699-711, 1992.
- Uralil, F.S., Newaz, G.M., and Lustiger A., "Processing Effects and Damage Tolerance in PEEK Composites", appeared in the <u>J. of Polymer Composites</u>, 12: 35-51, (1991).
- Lustiger A., and Newaz, G.M., "Interlaminar Failure and Craze Growth in PEEK Composites Under Cyclic Loading", <u>J. of Composite Materials</u>, Vol. 24, 175-187, Feb. 1990.
- Lustiger, A., and Newaz, G.M., "Processing and Structural Optimization of PEEK Composites: Mechanical Response and Morphology", <u>Journal of</u> <u>Polymer Composites</u>, Vol. 11, #1, 65-75, (1990).
- Newaz, G.M. and Mall S., "Relaxation-Controlled Delamination Growth in Advanced Thermoset and Thermoplastic Composites Under Fatigue Loading at Elevated Temperature", <u>Journal of Composite Materials</u>, Vol. 23, #2, 133-145, (1989).
- Newaz, G.M., and Walsh, W.J., "Interrelationship of Damage and Strain in Particulate Composites", <u>J. of Composite Materials</u>, Vol. 23, #4, 326-336, (1989).
- 20. Newaz, G.M., and Ahmad, J., "A Simple Technique for Measuring Mode I Delamination Energy Release Rate in Polymetric Composites", <u>Engineering</u> <u>Fracture Mechanics</u>, Vol. 3, #4, 541-552, (1989).

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- E. Paper Published in Conference Proceedings

Refereed Papers

- 1. Newaz, G.M., "Compressive Response of MMC", Durability of Composite Marterials, Editor, R.C. Wethrehold, MD., Vol. 51, American Society of Mechanical Engineers, 13-28, (1995).
- 2. Newaz, G.M., Brust, F.W., Jarmon, D.C., and Cairo, R.R., "Mechanical Response Modeling Dual Fiber CMC", to be presented at the American Ceramic Society Annual Meeting, Cocoa Beach, Florida, January 12-14, 1994.
- 3. Newaz, G.M., and Majumdar, B.S., "Deformation and Damage Mechanisms in MMC Under Isothermal and TMF Loading", presented at the ASTM Symposium, Montreal, May 16-17, 1994.
- Newaz, G.M., Majumdar, B.S., and Brust, F.W., "Inelastic Deformation Mechanisms in Ti 15-3/SCS-6 Lamina Under Compression", Invited Paper, ASTM Symposium on Life Prediction of Titanium Composites" (to appear in an ASTM STP), Hilton Head, S. Carolina, March 22-24, 1994.
- 5. Newaz, G.M., "Evaluation and Modeling of Mechanical Response and Strength of MMC in Compression", presented at the Air Force Mechanics Composites Review Meeting, Dayton, OH, Dec. 7-8, 1993.
- 6. Newaz, G.M., "Inelastic Response of Off-Axis MMC Lamina", presented at the 6th Meeting of National Institute for Mechanics and Life Prediction of Composites (NIC), Cleveland, OH, November 3, 1993.
- 7. Newaz, G.M., "Deformation and Failure of High Temperature Metal Matrix Composites", Keynote Speaker, Italian Group of Fracture (IGF9), Rome, Italy, June 1993.
- Newaz, G.M., "Compression Behavior of Unidirectional MMC", Invited Paper, Air Force, Titanium Metal Matrix Composite Workshop II, LaJolla, CA, June 1993.
- 9. Newaz, G.M., "Comparison of Inelastic Deformation in Transverse Lamina under Tension and Compression", Invited Lecture in MEET'N 93-Joint ASME-ASCE-SEM Meeting, Charlottesville, VA, June 7-8, 1993.

- Newaz, G.M., and Majumdar, B.S., "Damage and Plasticity in MMC: Implications on Constitutive Modeling", proceedings of the Ninth International Conference on Composite Materials (ICCM-9), Madrid, Spain, July 12-16, 1993.
- Sierakowski, R. and Newaz, G.M., "Damage Tolerance in Advanced Composites: A Review of Key Elements", proceedings of the 8th American Society for Composites Annual Conference, Cleveland, OH, October 17-19, 1993.
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IV. <u>SERVICE</u>

- A. Administrative Appointments at WSU: N/A
- B. Administrative Appointments at Other College/Universities: N/A
- C. Committee Assignments in Last Five Years: N/A
- D. Positions Held in Professional Associations: None
- E. Membership in Private or Public Agencies Related to Discipline: None
- F. Professional Consultation

Lockheed	1990-present
US Navy	1989-1994
Pratt & Whitney	1992-present
Sealy	1994

- G. Journal/Editorial Activity Editorial Board-ASTM Journal of Testing Evaluation Editorial Board-Advanced Performance Materials, Kluwer Publications
- H. Other Professional Related Service:

Member:	ASME Materials Division/Symposia Organizer
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Analysis of Quasi-Isotropic MMC Using Iosipescu Shear Test

K. Zhang and G. M. Newaz Mechanical Engineering Department Wayne State University Detroit, Michigan and S. Chandu and J. Ahmad Research Applications, Inc. Dayton, Ohio

ABSTRACT

The shear constitutive response and deformation mechanisms under Iosipescu test for a quasi-isotropic, [0/+45/-45/90]_s laminate made from TMIETAL 21S/SCS-6 MIMC were investigated using a combination of mechanical measurement, microstructure analysis and finite element method. The objectives were to evaluate the material response using the Iosipescu test and attempt to establish Iosipescu test as a reasonable routine measurement for properties under shearing loading for Quasi-isotropic MIMCs. Shear induced inelastic deformation is dominant due to plasticity which is associated with slip bands and damage which includes matrix grain boundary cracks, reaction zone cracks, debonding and fiber cracks. The prediction for the constitutive response using finite element analysis fits test data very well. The correlation of predicted results with experimental data shows that Iosipescu shear test is a valid method to investigate shear properties of quasi-isotropic MMC laminates.

INTRODUCTION

Considerable research to date has been conducted in characterizing silicon-carbide (SiC) fiber-reinforced titanium composites to understand their monotonic behavior (Sun and Chen 1989; Pindera, 1989; Sun *et al.* 1990; Newaz and Majumdar, 1991; Majumdar and Newaz, 1992; Newaz and Majumdar, 1993; Brust, Majumdar and Newaz, 1993; Newaz and Majumdar, 1994; Gao and Zhao, 1995; Allen Jones and Boyd, 1994; Newaz, Majumdar and Brust, 1996, Newaz and Zhang, 1996). One important aspect is a continuing effort to understand and model the composite's constitutive response and failure conditions.

Constitutive modeling and inelastic deformation mechanisms have been studied extensively for longitudinal and transverse MMC laminates. However, little attention has been paid to investigate the constitutive response of pure shearing loading on MMCs, especially for the differently oriented ply-stacked laminate, its inelastic deformation mechanisms and the evolution of damage to failure. Furthermore, properties and constitutive response under pure shearing loading for MMCs using a reliable test technique has not been identified to date.

A variety of test methods are available for introducing shear stresses in unidirectional

fiber MMCs. However, all of the tests have inherent deficiencies or disadvantages. The most uniform shear strain-stress state can be achieved in a MMC by applying torsional loading to a thin-walled tube specimen. However, such specimens are expensive requiring special fixtures and process techniques. The Iosipescu Test standard as explained in ASTM D 5379M-93 is recommended for in-plane shear testing specimens with unidirectional, cross-ply, woven composite and short-fiber composites. Tests on MMCs of continuous fibers are not within the scope of the standard. Iosipescu test has been conducted with various composites (Adams and Lewis 1995; Pindera, Ifju and Post, 1990; Swanson, Messick and Toombes, 1985; Morton *et al.*, 1992). For continuous fiber MMCs, especially oriented ply-stacked MMCs, some limited analysis has been performed as well (Santhosh and Ahmad, 1992; Chandu, Ahmad and Newaz, 1997).

For metal matrix composites, the tubular specimens can be quite expensive. Testing of tubes also requires specially designed fixtures (Swanson, Messick and Toombes, 1985). Offaxis tensile tests are not ideal for generating shear properties for titanium based metal matrix composites because of the relatively strong interaction of the transverse normal stress and shear stress at all possible angles due to the nature of orthotropy in a single ply. Also, off-axis tests are not applicable for multi-ply oriented laminates. The most reasonable approaches for shear testing of MMCs appear to be the Iosipescu test and $[\pm 45]_s$ tension test. The specific advantage of the Iosipescu shear test is the ability to develop pure shear stresses in a line zone between the two notches in the specimen. For ± 45 tension test, the shear properties are estimated after manipulating tension strain data in the normal and transverse directions. In this test, the plies in the lamina are subjected to both transverse normal and shear stresses.

EXPERIEMENTAL APPROACH

The material for testing was Ti 15-3/SCS-6 composite of 8-ply unidirectional for off-axis tension test and 8-ply quasi-isotropic $[0/+45/-45/90]_s$ lay-up for Iosipescu shear tests, approximately 0.00785-inch thick each ply with a fiber volume fraction of approximately 0.34. The SCS-6 (SiC) fiber diameter is approximately 140 μ m.

The test specimens were cut from an electric-discharge machining (EDM) technique and then mechanically polished after EDM machining to remove any damage associated with the machining. Monotonic loading was applied at room temperature. All specimens were tested in the as-fabricated condition, i.e., no heat-treatment was performed prior to the testing.

The dimensions of a specimen with a test fixture and a strain gage (Micro-measurements, A2P-08-C085C-500) designed for pure shear strain measurement is shown in figure 1. The tests were performed at MTS test machine with an Iosipescu fixture. The strain data came from the strain gage and the stress data from the loading value divided by the cross section area between the notch tips. The specimens were loaded to a different deformation levels then unloaded to zero to investigate the constitutive response in both elastic and inelastic regimes and to determine how consistent are the elastic shear modulus, yielding and hardening tendency. Unloading from different loading levels, the zero-load strain offset, plasticity versa damage effects in nonlinear regime can be analyzed. In order to obtain information about shear failure of 8-ply quasi-isotropic $[0/+45/-45/90]_s$ Ti 15-3/SCS-6 composite, one of the specimens was loaded until its



Figure 1. Dimensions of an Iosipescu test specimen with strain gage under loading fixture.

failure. Following mechanical testing, specimens were sectioned using a diamond wafering blade; debonding and fiber cracking can be significant during machining if adequate care is not taken during sectioning. Specimens were metallographically polished up to the first set of fibers. Specimens were etched using Kroll's reagent, which was found to be effective in revealing matrix damage and slip bands

RESULTS AND DISCUSSION

MECHANICAL BEHAVIOR

TABLE I shows the Iosipescu test results for specimens OH3-2, OH3-4,OH3-8 and OH3-12. It can be seen that the apparent yield stress was in the range of 200 MPa to 270 MPa and the corresponding yielding strain ranged from 0.0038 to 0.005.

Specimen Number	Elastic Shear Modulus (GPa)	Maximum Stress (MPa)	Maximum Strain	Apparent Yield Stress (MPa)	Apparent Yield Strain	Unload Strain Offset	Failure Stress (MPa) / Failure Strain
OH3-2	55	757	0.021	270	0.0045	х	757/0.021
OH3-4	48	434	0.01	250	0.005	0.0004	х
ОН3-8	54	559	0.014	200	0.0038	0.0017	х
OH3-12	48	659	0.017	250	0.005	0.0028	Х

TABLE I IOSIPESCU SHEAR TEST RESULTS

The elastic shear modulus are within 48 MPa to 55 MPa. Specimen OH3-2 failed with notched region pure shear stress reaching 757 MPa and strain reaching to 0.021.

Figure 2 shows the shear response for these specimens. For specimen OH3-4 the maximum shear stress was 434 MPa with maximum shear strain reaching 0.01. The specimen then was unloaded to zero. The curve shows that (a) there was no obvious turning point between elastic and inelastic regimes though the curve slope began decreasing gradually after about 250MPa. This was also the case for the other specimens; (b) the unloading slope decreased and zero-load strain offset are due to associated damage and plasticity respectively. The nonlinearity of the unloading curve near zero-load might be caused by damage closure. Figure 3 is a photo-micrograph of etched specimen OH3-4 in the notched zone (strain gage covered zone). There were apparent cracks along grain boundaries and the fiber and matrix bonding reaction zone. Stiffness loss experienced in the unloading curve is due to damage in the net section area of the specimen.

For specimen OH3-8, the maximum shear stress applied was 559 MPa with corresponding maximum shear strain up to 0.014. The specimen was then unloaded to zero. The curve shows a consistent tendency as that of OH3-4 except in the unloading period. The zero-load strain offset means that plasticity took a dominant role in the inelastic deformation regime. Figure 4 illustrates obvious matrix slip bands in etched OH3-8 specimen. These slip bands are associated with the plastic deformation mechanism.



Figure 2. Mechanical response for Iosipescu test specimens of Quasi-isotropic laminate made of TMIETAL 21S/SCS-6 MMC.



Figure 3. Etched microstructure (x500) of specimen OH3-4 in strain gage covered zone, cracks along grain boundaries and bonding reaction zone.



Figure 4. Etched microstructure (x1000) of specimen OH3-8, slip bands intiated from grain boundaries and bonding reaction zones.

CONSTITUTIVE RESPONSE MODELING

For the $[0/+45/-45/90]_s$ laminate, each laminae of the TMIETAL 21S/SCS-6 MMC is treated as an anisotropic ply. The laminate properties depend on the individual plies. In the model, each ply is modeled as a separate layer and connected to other plies through common nodes. The model is based on a plane stress analysis. In the inelastic range, the deformation becomes much more complex and the hardening effects are significant. It is necessary to determine inelastic equivalent stress, σ_e , like what we have in the case of homogenous plastic deformation. With a formulation regarding σ_e , we can relate our test data (monotonic tension test, for instance) to our analysis. Here we have according to Sun (1989):

$$\sigma_{\rm e} = \left[\frac{3}{2}(\sigma_{22}^2 + 2 a_{66} \sigma_{12}^2)\right]^{1/2} \tag{1}$$

where, σ_e represents equivalent inelastic stress and 1 represents fiber direction, 2 is 90° of the fiber direction (transverse). σ_{22} represents the transverse stress of a MMC laminae. σ_{12} represents the in-plane shear stress. σ_{11} represents stress in the fiber direction.

Sun (Sun *et al.*, 1990) assumed that only the transverse normal stress σ_{22} and in planeshear stress σ_{12} of composite contribute to inelastic deformation. Whereas the longitudinal (fiber direction) σ_{11} stress does not affect inelastic deformation though it contributes to elastic deformation. This is in fact not true. The nonlinear deformation starts after the tension stress reached approximately 1100 MPa (Majumdar and Newaz 1992). But here we can take Sun's assumption as an approximation to simplify our calculation. With Sun's equation if we relate it to the 90° tension test data (Newaz and Zhang, 1997) as yield and hardening reference in an analysis, then for this situation (transverse loading), $\sigma_{12}=0$, and Sun's formula becomes:

$$\sigma_{\rm e} = \left[\frac{3}{2}\sigma_{22}^2\right]^{1/2} \tag{2}$$

This case, the apparent yield stress is

$$\sigma_{\rm e} = \sigma_{\rm y} = 250 \text{ MPa (test data)}$$
(3)

For off axis monotonic tension test.

$$\sigma_{11} = \sigma_x \cos^2 \theta \tag{4}$$

$$\sigma_{22} = \sigma_x \cos \theta \sin \theta \tag{5}$$

$$\sigma_{12} = \sigma_x \sin^2 \theta \tag{6}$$

at $\theta = 10^{\circ}$ we have

$$\sigma_{11} = 0.970\sigma_{\rm x} \tag{7}$$

$$\sigma_{22} = 0.030\sigma_{\rm x} \tag{8}$$

$$\sigma_{12} = -0.171\sigma_{\mathrm{x}} \tag{9}$$

Putting these value into Sun's formula, we get:

$$\sigma_{\rm e} = \left[\frac{3}{2}((0.970\sigma x)^2 + 2 a_{66}(-0.171\sigma x)^2)\right]^{1/2}$$
(10)

where x is loading direction, and θ is the angle between fiber direction and loading direction.

From the test data (Newaz and Zhang, 1997), the apparent yield stress for 10° tension loading at x direction is $\sigma_x = 850$ MPa. Whereas with transverse tension test we already know that yielding equivalent stress is 250 MPa. With this information, we can get a_{66} which is approximately about 1.46. For MMC inelastic deformation includes not only plastic but damage - matrix grain boundary cracks, fiber and matrix bond separation and fiber crackseffect too. Therefore, we should take both effects into the account in determining parameter a₆₆. We can modify Sun's model by choosing a suitable value of a₆₆. In Sun's paper (Sun et al., 1990) $a_{66} = 0.5$ was chosen to fit the yield stress test curve of different angles of off-axis monotonic tension tests for SCS-6/Ti-6-4 MMC. But for [0/+45/-45/90]s laminate, shear test model in which the plies stacked have different orientation, the situation is more complicated. Analysis also shows that in different elements of the model either transverse tension stresses or transverse compression stresses is in effect in addition to shear stresses. And different stress state combination has different yield and hardening condition. From test data the transverse compression yield stress (750 MPa in Newaz, Majumdar and Brust, 1996) is much higher than the transverse tension yield stress (250 MPa in Newaz and Zhang, 1997). Here we can still use equation (1) for both tension and compression occasions, but choose $a_{66} = 0.4$, $\sigma_y = 250$ MPa (yield) for a case when $\sigma 22 > 0$ in an element; choose $b_{66} = 0.5$, $\sigma_y = 750$ MPa (yield) for a case when $\sigma 22 < 0$ in an element. During the analysis we use FORTRAN subroutine to check each element in different plies to see whether $\sigma 22 > 0$ or $\sigma 22 < 0$ to determine the field parameter and then apply tension hardening condition or compression hardening condition to the element accordingly. We used the transverse (90°) monotonic tension and compression test data as the hardening reference for the analysis.

FINITE ELEMENT ANALYSIS RESULTS

We used ABAQUS code to simulate a monotonic loading Iosipescu shear test for TMIETAL 21S/SCS-6 MMC [0/+45/-45/90]_s laminate. Figure 5 shows the comparison of the constitutive responses between the FEA predication and the tested specimen OH3-8. The calculated response comes from the center portion elements of the notch zone (strain gage covered zone) of the surface layer, it is found that if the elements were taken from the 80% of the central portion between two notch tips, then the calculated curves can fit the test results well, otherwise the calculated response may be lower than the measured value. It can be seen that the calculated result fits the test data very well. Unlike the testing response, the calculated unloading stiffness, remained the same as the loading one. This is because the FEA model did



Figure 5. Comparison of analysis prediction and test measured constitutive shear response for Quasi-isotopic laminate made of the TMIETAL 21S/SCS-6 MMC.

not take the damage closure effect into the unloading analysis.

With the model surface color stress contours (they can not be clearly shown in white and black) for σ_x and τ_{xy} (x coincident with fiber direction) show that there is a small red color stress concentrated area on the edge next to a notch tip, its location is approximately the same as it was mentioned previously which caused the surface fiber fractures in specimen OH3-4 and further initiated failure in specimen OH3-2. The color contour also shows σ_x along fiber direction within the area exceeded 1900 MPa, Referring to Majumdar and Newaz, 1991, specimen under 0° tension loading failure at about 1500 MPa to 1800 MPa. This can explain why there were fiber fractures in the area of the surface layer of specimen OH3-4 and OH3-8.

It should be pointed out that Sun did not take the fiber direction inelastic deformation and neither the damage effects into his model. Therefore, when finite element model for MMC based on this assumption is limited to shear strain < 0.01, the calculation may provide good prediction. Errors may increase when the shear strain is larger than this limit.

CONCLUSIONS

1. Iosipescu shear test provides a reasonable method for measurement of shear constitutive response of MMC material.

2. Iosipescu shear test has very good reproducibility in terms of overall properties and mechanical behavior. Iosipescu test can be recommended as a routine measurement to obtain MMC material linear and nonlinear shear properties at room temperature. Finite element analysis model for MMC Iosipescu test using Sun's assumption as an approximation plus a subroutine to check the stress state to determine the transverse tension or compression stress and then applying different yielding and hardening criterion can obtain satisfactory result for the case of shear strain<0.01.

3. Sun's effective stress model for plastic deformation was successfully used for prediction of experimental data. The basic input for finite element analysis required 0° tension, 90° transverse tension and compression data. Shearing coefficient a_{66} was assumed to be generated by manipulating off-axis tension test data. However a_{66} obtained by this way included neither damage effect nor fiber direction inelastic effect, and the subsequently determined a_{66} can not perform a good enough prediction in the analysis. Furthermore one parameter a_{66} is not enough to interpret the difference between transverse tension and compression constitutive responses. So a better way is to assume two trial shearing parameter a_{66} and b_{66} to interpret transverse tension and transverse compression of a laminae respectively. The values of a_{66} and b_{66} are dependent of the property of the material.

4. At an early stage of shear inelastic deformation (strain <0.01), damage effect play an important role. This can be seen by the compliance change in the unloading curve. The damage mechanism was associated with matrix grain boundary crack and cracks along the fiber and matrix reaction zone. When shear deformation increases, plasticity becomes a primary inelastic deformation mechanism which is associated with slip bands in the matrix.

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ANALYSIS OF INTERFACIAL CRACKS IN A TBC/SUPERALLOY SYSTEM UNDER THERMO-MECHANICAL LOADING

S. Q. Nusier and G. M. Newaz¹ Mechanical Engineering Department Wayne State University Detroit, Michigan 48202

ABSTRACT

In thermal barrier coatings (TBC) residual stresses develop during cool down from processing temperature due to the thermal expansion mismatch between the different layers (substrate, bond coat, and TBC). These residual stresses can initiate microcracks at the bond coat/TBC interface and can lead to debonding at the bond coat/TBC interface. The effect of voids or crack like flaws at the interface can be responsible for initiating debonding and accelerate the oxidation process. Effect of oxidation growth between bond coat and the ceramic layer (TBC) can be modeled as volume increase. In this work we represent this change in volume as an induced pressure across the interface. Mixed-mode fracture analysis of a thin circular delamination in an-axisymmetrically multi-layer circular plate is developed. Geometrical nonlinearity is included in the analysis, since we have a large deflection case. The elastic deformation problem of a circular plate subjected to a clamped boundary condition at the edge delamination, an out of plane pressure load, and a compressive stress due to thermal mismatch between different layers, was solved numerically using a Rayleigh-Ritz method. The strain energy release rate was evaluated by means of the path-independent J-integral. The numerical results of this problem based on the energy method were verified using the finite element method. Both method correlate well in predicting the energy release rate for Mode I and Mode II, deflection, and postbuckling solutions. The energy release rate G, for both Mode I and Mode II using virtual crack extension method were evaluated. The specimen was cooled down from processing temperature of 1000 °C to 0 °C. The variation of the properties as a function of temperature were used for analysis. It was found that the use of temperature dependent properties in contrast to constant properties provide significantly different values of J-integral and G.

INTRODUCTION

Thermal barrier coatings (TBCs) provide thermal insulation and the bond coat provides oxidation resistance at high temperature to high temperature alloy substrates. Plasma-sprayed zirconia-yttria ceramic layer with a nickel-chromium-aluminum-yttrium bond coat on a substrate made of nickel-based superalloy (Chang, et al., 1987) is a common superalloy/TBC system. Application of these superalloy/TBC systems can be found in both aerospace and land-based gas turbine engines. In automotive applications, the piston head for diesel engine is coated to achieve longer life time and higher performance in terms of fuel reduction and power. However, these coatings have durability problems, due to the material and thermal mismatch between the coating and the metallic substrate. Thermal residual stresses develop during cool down from processing temperatures in TBC/metallic substrate. Environmental effects, specifically oxidation, create additional residual stresses due to the growth of an oxide layer causing additional material mismatch between the oxide surface and the TBC. These

¹Author receiving all communication
residual stresses may initiate microcracks such as debonding and radial cracks and can have profound effect on the response of the TBC and interfacial damage accumulation and failure. Their understanding is essential to predict the behavior of the coatings and their performance. The processing technique itself may produce voids or elongated flaws such as air bubble along the interfaces, which may initiate debonding.

Thermal fracture of multilayer ceramic thermal barrier coatings was studied by Takeuchi and Kokini (1994). Controlled experiments along with finite element modeling were used. Their work showed that surface cracks are formed as a result of tensile stresses that are created following stress relaxation in the TBC at steady-state high temperatures. The effect of a transient thermal load on a coating which is bonded to a cylindrical substrate was studied by Hornack and Kokini (1988). Finite element method was used to obtain a solution for a circumferential edge crack normal to the coating. Their analysis showed that smaller heat transfer rates result in smaller stress intensity factors. The finite element method has been used in conjunction with a numerical interface fracture mechanics model to investigate the structural response of coated brittle materials subjected to normal and shear loads, by Oneil and Wayne (1994). They examined how flaw orientation affects crack propagation through the coating, interface, and substrate. A comparison between the uncoated and coated substrate has shown that cracks are strongly influenced toward interfaces because of the preferred release of critical strain energy. Interfacial fracture in bimaterials often is due to a combination of Mode I and Mode II crack growth. A finite element model to calculate the Mode II stress-intensity factors was developed by Van der Zande and Grootenboer (1986). The optimum size for a so-called singular element has been determined. Ahmad (1993) provides micromechanics based fracture analysis and correlation of experimental data for metal-ceramic and other interfaces. Suo (1995) studied wrinkles which induce interfacial stress and cause voiding. A linear bifurcation analysis has been carried out when wrinkles just start. The oxide scale thickens slowly at a higher temperature, until the compressive stress generated by oxidation and cooling causes it to spall off. The strain energy release rate components G_{I} and G_{II} in Mode I and Mode II at the tip of an interface crack in a bimaterial plate under tension in a direction normal to the interface were evaluated using finite element analysis and modified crack closure integral (MCCI) technique by Dattagurn, et al., (1994). The finite element results for all the models show increasing Mode II dominance as $\Delta a \rightarrow 0$. An elastoplastic solution for the interface crack with contact zones was studied by Aravas and Sharma (1991). It was shown that the elastic asymptotic solutions based on the assumption of a closed crack tip predicted material interpenetration. Also, it was shown that the asymptotic solution is separable in r and θ at the crack tip. Singular thermal stress fields in bonded viscoelastic quarter planes are studied by Blanchard and Ghoniem (1989). It was shown that the order of the singularity generally increases with time. Singular stress and heat flux fields at the tip of the crack in a general nonhomogeneous material are studied by Jin and Noda (1994). It was found that the crack-tip field singularities and angular distributions are the same as those in the homogeneous material. New domain integrals for axisymmetric interface crack problems are derived by Nahta and Moran (1993). The effect of crack front curvature is shown to play an important role in the derivation of the integrals.

Descriptions of residual stresses and their influence on mechanical failure of the coating were studied by Evans, et al., (1983). The mechanics of the delamination and spalling of compressed films or coatings has been analyzed using a combination of fracture mechanics and post-buckling theory by Evans and Hutchinson (1984). The phenomenon of delamination buckling and growth in a time dependent radial compressive load is analyzed by Boltega and Maewal (1983). An iterative procedure based on the fourth-order Runge-Kutta integration formula is used to generate a family of nondimensionalized postbuckling solutions of von Karman's nonlinear plate theory by Yin (1985). A mixed-mode fracture analysis combining nonlinear thin-plate stress solutions with crack-tip elasticity results has been developed to account for local variations of G_{II} , G_{II} , and G_{III} in thin-film debond problems associated

with large film deformations by Chai (1990). A shaft-loaded blister test has been developed by Wan and Mai (1995) to measure the interfacial energy of a thin flexible polymeric film adhered to a rigid substarte. A theoretical analysis is given of an axisymmetric debond ('blister'). Expressions have been derived which describe the critical stress and pressure necessary to rupture oxide blisters which form on aluminum during growth of corrosion pits by Ryan and McCafferty (1995).

Although numerous effects have been made to an understanding of the effect of cracks on the life of TBC coated specimens require special attention. It is now determined that in some TBC systems such as electron beam-plasma vapor deposition (EB-PVD), there is microcrack initiation that coalesce to form major delamination cracks as reported by Newaz, et. al., (1996). Interfacial crack in a stepped-disk specimen is shown in Fig. 1. An important consideration is the nature of crack growth characteristics at the TBC/bond coat interface. In a previous study by Nusier and Newaz (1996), it was shown that a central delamination under pure thermal loading has no stress intensification at the crack tip unless the delamination is large enough to promote buckling. By investigating the issue of interfacial crack growth, we will be able to evaluate the condition necessary for their growth under thermo-mechanical loading. This button specimen under consideration is amenable to axisymmetric modeling due to geometry. The importance of this problem is due the fact that in order to achieve realistic prediction of TBC spallation performance, one needs to study the interaction of various layers and interfacial cracks at high temperature.

THEORETICAL AND COMPUTATIONAL ANALYSIS

The TBC coating in a button specimen as studied by Nusier and Newaz (1995) is in a state of biaxial compression. Residual compression stresses has been observed in TBC coating that was applied using Electron Beam-Plasma Vapor Deposition (EB-PVD) technique. This resdiual compression stress arise because of thermal expansion mismatch. Buckling failure mode has been observed by Newaz, et al. (1996) in the EB-PVD system. Oxidation growth between bond coat and ceramic layer (TBC) can be modeled as volume increase which can be represented as an induced pressure across the interface (TBC/Oxide). Mixed-mode fracture analysis of a thin circular delamination in an-axisymmetrically multi layer circular plate is given as follows.

Theoretical Analysis

Let a circular plate of radius *a* be clamped at the edge of the delimination and subject to a uniformly distributed pressure *p*. The clamped edge has a radial displacement $a\mathcal{E}_0$ due to the applied compressive load. The deflection and slope at the clamped edge is zero, also the radial displacement at the center of the plate is zero. For a large deflection, the strain in the radial direction is

$$\varepsilon_{\rm r} = \frac{{\rm d} {\rm u}}{{\rm d} {\rm r}} + \frac{1}{2} (\frac{{\rm d} {\rm w}}{{\rm d} {\rm r}})^2$$

and the strain in the tangential direction is

(1)

(2)

 $\epsilon_{\theta} = \frac{u}{r}$

where u and w are the radial and vertical components of the displacement vector, respectively. Let N_r and N_{θ} be the corresponding tensile forces per unit length and applying Hooke's law, we obtain

$$N_{r} = \frac{Eh}{1 - v^{2}} (\varepsilon_{r} + v\varepsilon_{\theta})$$
$$N_{\theta} = \frac{Eh}{1 - v^{2}} (\varepsilon_{\theta} + v\varepsilon_{r})$$

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(3)

where h is the plate thickness. The strain energy due to bending is given by

$$V = \frac{D}{2} \int_{0}^{2\pi} \int_{0}^{a} \left[\left(\frac{\partial^{2} w}{\partial r^{2}} \right)^{2} + \frac{1}{r^{2}} \left(\frac{\partial w}{\partial r} \right)^{2} + \frac{2\nu}{r} \frac{\partial w}{\partial r} \frac{\partial^{2} w}{\partial r^{2}} \right] r dr d\theta$$
(4)

where D is the flexural rigidity of the plate, and is given by

$$D = \frac{Eh^3}{12(1 - v^2)}$$
(5)

The strain energy due to stretching of the middle plane is

$$V_{1} = 2\pi \int_{0}^{a} \left(\frac{N_{r}\varepsilon_{r}}{2} + \frac{N_{\theta}\varepsilon_{\theta}}{2}\right) r dr$$
(6)

The elastic deformation problem for a circular plate subjected to the boundary conditions indicated earlier was solved using a Rayleigh-Ritz method (an energy method) based on the following polynomial series solution

$$w = \sum_{i=1}^{n} c_i (1 - \frac{r^2}{a^2})^{i+1}, \text{ and}$$
$$u = r(a^2 - r^2) (\sum_{i=1}^{n} b_i r^{2i-2}) - \varepsilon_0 r$$

(7)

where c_i and b_i are constants to be evaluated from the condition that the total energy of the plate for a position of equilibrium is a minimum. Hence,

$$\frac{\partial V_{i}}{\partial b_{i}} = 0, \quad \text{and} \\ \frac{\partial (V + V_{i})}{\partial c_{i}} \delta c_{i} = 2\pi \int_{0}^{a} p \delta wr dr$$
(8)

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The first part of Eqn. 8 give us an n-linear equations for the constants b_i , these equations are solved in symbolic form, the second part of Eqn. 8 give us an n-nonlinear equations for the constants C_i , the solution of the constants b_i were used. All of this symbolic calculation are carried out by using Mathematica (1996). The set of nonlinear equations have been solved by IMSL (1989) library subroutine NEQNJ based on Levenberg -Marquardt algorithm with a user-supplied jacobian obtain by Mathematica also. The solution converged after 6 terms (n=6) for the case considered.

The compressive strain in the TBC layer can be obtained by applying the equilibrium radial force equation for the three layers. This solution is valid only for thick substrate. The radial stress in each layer is written in the following form

$$\sigma^{c} = \frac{E^{c}}{1 - \nu^{c}} [\varepsilon - \alpha^{c} (T - T_{r})]$$

$$\sigma^{b} = \frac{E^{b}}{1 - \nu^{b}} [\varepsilon - \alpha^{b} (T - T_{r})]$$

$$\sigma^{s} = \frac{E^{s}}{1 - \nu^{s}} [\varepsilon - \alpha^{s} (T - T_{r})]$$
(9)

where σ is the radial stress, α is the thermal expansion coefficient, and T_r is the stress free temperature, the superscript *c*, *b*, and *s* refers to TBC, bond coat and substrate, respectively. The equilibrium radial force equation for the three layers is

$$\sum F = \sigma^{c} A^{c} + \sigma^{b} A^{b} + \sigma^{s} A^{s} = 0$$
(10)

Substituting Eqn. 9 into Eqn. 10, and solving for ε gives

$$\varepsilon = \frac{\left(\frac{E^{c}}{1-\nu^{c}}\alpha^{c}A^{c} + \frac{E^{b}}{1-\nu^{b}}\alpha^{b}A^{b} + \frac{E^{s}}{1-\nu^{s}}\alpha^{s}A^{s}\right)(T-T_{r})}{\frac{E^{c}}{1-\nu^{c}}A^{c} + \frac{E^{b}}{1-\nu^{b}}A^{b} + \frac{E^{s}}{1-\nu^{s}}A^{s}}$$
(11)

Now, the compressive strain in TBC is

$$\varepsilon_0 = \varepsilon - \alpha^{c} (T - T_r)$$
⁽¹²⁾

In case of temperature dependent properties, the previous Eqns. 9-12 will be written in incremental form for each dT, then they will add up to get the right value of ε . For example, the variation of ε due to dT change in temperature will be given as

$$d\varepsilon = \alpha |_{T} (T - T_{0}) - \alpha |_{T - dT} (T - dT - T_{0})$$
(13)

where T_0 is the reference temperature and is equal to zero.

Computational Fracture Analysis

Linear elastic fracture mechanics can be used to asses the conditions for crack growth of bimaterial interfaces. The mechanics of interface fracture can be traced back to the earlier works of Griffith (1921) and Irwin (1960) on the general theory of fracture, of Williams (1959) on the elastic stress distribution around an interface crack, of England (1965), Erodogan (1965), and Rice and Sih (1965) on explicit solutions for interface cracks. The classical fracture mechanics concepts enable us to predict, without a detailed description of the crack tip processes, crack growth behavior in a fracture specimen. In recent years, complexity of obtaining closed-form solutions have been circumvented through computational fracture analysis to determine conditions for crack growth at bimaterial interfaces. However, the analytical basis is essential to study the critical parameters that characterize interface fracture.

With the interface on the x_1 -axis, let E_1 , μ_1 and ν_1 be the Young's modulus, shear modulus and Poisson's ratio of material 1 lying above the interface ($x_2 > 0$) with similar quantities, E_2 , μ_2 and ν_2 , for material 2 lying below the interface. For plane problems with traction boundary conditions, only two nondimensional combinations of the four independent material moduli parameters enter into any solution as discussed by Dunders (1969). For plane strain, the moduli mismatch parameters of Dundurs are

$$\alpha = \frac{\overline{E}_1 - \overline{E}_2}{\overline{E}_1 + \overline{E}_2} \quad \text{and} \quad \beta = \frac{\mu_1 (1 - 2\nu_2) - \mu_2 (1 - 2\nu_1)}{\mu_1 (1 - \nu_2) + \mu_2 (1 - \nu_1)}$$
(14)

where $\overline{E} = E / (1 - v^2)$ is the plane strain tensile modulus. Note that α and β both vanish when dissimilarity between the elastic properties of the materials is absent, and α and β change signs when the materials are switched. For each material pair, a universal singular crack tip field exists at the crack tip according to linear elasticity theory for a traction-free line crack. For the plane problems, the normal and shear stresses of the singular field acting on the interface a distance r ahead of the tip can be written according to the "function" form

$$\sigma_{22} + i\sigma_{12} = \frac{(K_1 + iK_2)r^{i\varepsilon}}{\sqrt{2\pi r}}$$
⁽¹⁵⁾

where $i = \sqrt{-1}$ and the oscillation index ε depends on β according to

$$\varepsilon = \frac{1}{2\pi} \ln \left(\frac{1 - \beta}{1 + \beta} \right)$$
(16)

In plane strain, the crack face displacements a distance r behind the tip, $\delta_i = u_i(-r,0^+) - u_i(-r,0^-)$, are given by

$$\delta_{2} + i\delta_{1} = \frac{4}{\sqrt{2\pi}} \frac{(1/\overline{E}_{1} + 1/\overline{E}_{2})}{(1+2i\varepsilon)\cosh(\pi\varepsilon)} (K_{1} + iK_{2})\sqrt{r} r^{i\varepsilon}$$
(17)

The amplitude factors, K_1 and K_2 , depends linearly on the applied loads and on the details of the full geometry of the body, as will be illustrated below. These stress intensity factors are defined to be consistent with corresponding stress intensity factors for cracks in homogeneous problems (Rice, 1988). The energy release rate per unit length of extension of the crack in the interface for plane strain is related to the stress intensity factors by

$$G = \frac{(1/\overline{E}_1 + 1/\overline{E}_2)}{2\cosh^2(\pi\epsilon)} (K_1^2 + iK_2^2)$$
(18)

which is the generalization of Irwin's result for a homogeneous isotropic material. When $\varepsilon \neq 0$, the relative proportion of normal and shear stresses on the interface in the singular field varies slowly according to $r^{i\varepsilon}$, and this feature complicates the implementation of interfacial mechanics in several respects. When $\varepsilon \neq 0$, the traction-free line crack solution is not fully consistent since the solution (England, 1965) implies that the crack faces interpenetrate behind the tip.

Another approach to characterize fracture at bimaterial interface is via the J-integral. Originally developed by Eshelby (1956), the basic concept of J-integral is a path independent evaluation of the energy release rate. In other words, it is a measure of decrease in potential energy of the system with increase in crack length. In linear elastic fracture mechanics "J" is equivalent to G. The J-integral can be written as (Rice, 1968)

$$J = \int_{\Gamma} W dy - \int_{\Gamma} T \frac{\partial u}{\partial x} ds$$
(19)

where W is the strain energy density. Fig. 2 shows the notation and parameters for J-integral for a homogeneous medium. The analysis can be easily extended for a line crack between two materials assuming crack growth along the interface. The essence of J definition and its meaning remains unaltered for a crack at the bimaterial interface.

Following the work done by Yin (1985), the energy release rate associated with uniformexpansion growth of a circular delamination in a compressively loaded plate is obtained by means of the M-integral. The strain energy-release rate per unit increment of the area of delimination is given by

$$G = \frac{1 - v^2}{2Eh} \{ \left[\frac{Eh}{1 - v} \varepsilon_0 - N_r(a) \right]^2 + 12 \left[\frac{M_r(a)}{h} \right]^2 \}$$

where $M_r(r)$ is given by

$$M_{r}(r) = D(w'' + vw' / r)$$
(21)

(20)

where the prime denote differentiation with respect to r.

For arbitrary combinations of N and M the stress field at the crack tip is governed by both K_{I} and K_{II} (Thouless, et al., 1987). Dimensional considerations require that the stress intensity factors be related to the load quantities by

$$K_{I} = d_{1}Nh^{-0.5} + d_{2}Mh^{-1.5}$$

$$K_{II} = d_{3}Nh^{-0.5} + d_{4}Mh^{-1.5}$$
(22)

where the d_i are constants. The energy release rate is given by

$$G = \frac{(1 - v^2)}{E} (K_I^2 + K_{II}^2)$$
(23)

Solving the crack problem for one loading combination and comparison of Eqns. 22 and 23 gives (Thouless, et al., 1987)

$$d_1 = 0.434, \quad d_2 = 1.934, \quad d_3 = 0.558, \quad d_4 = -1.503$$
(24)

There is one idealized condition that we explored related to the specimen geometry. The presence of circumferential crack between the ceramic layer and the bond coat layer is as shown in Fig. 1 which was analyzed using finite element method. The general code ABAQUS (1995) was used for these analyses. The energy release rate G can be estimated by using the virtual crack extension method. The J-integral value can be found directly from ABAQUS.

Within linear elastic fracture mechanics two parameters are generally used to describe the conditions at the crack tip, normally the stress intensity factor(s) and the energy release rate. Evaluation of the stress intensity factor requires a through understanding of the state of stress at the crack tip. Energy release rate evaluation, on the other hand, is based more on an energy criteria and hence more popular.

A procedure for calculating the energy release rate, G, that has gained increasing acceptance over the past decade is the Virtual Crack Extension Method(VCEM). This was originally developed by Hellen and Parks (1975), who subsequently extended the method to cover material nonlinearities, and the evaluation of the J-integral. The method of VCE used to evaluate G is explained in the following few steps. Consider the following Fig. 3(a). The energy release rates in mode I and II are given by

$$G_{I} = \frac{1}{2A} f_{x} \Delta u_{x}$$
$$G_{II} = \frac{1}{2A} f_{y} \Delta u_{y}$$

Where f_x and f_y are the reaction forces, A is the crack surface area corresponding to Δa , and similarly Δu_x and Δu_y are the difference in the displacements of nodes A and B in the x and y directions respectively (Fig. 3(b)).

(25)

The reaction forces and the displacements were obtained via two runs. For the first run, we assume a very weak spring, so the opening displacement and sliding displacement can be determined from springs deflection. In the second run the spring stiffness is assumed to be very high compared to the material stiffness, so the reaction forces can be determined. In this study a eight node isoparametric element are used, the energy release rates in mode I and mode II are given by

$$G_{I} = \frac{1}{2A} [f_{x1} \Delta u_{x1} + f_{x2} \Delta u_{x2}]$$

$$G_{II} = \frac{1}{2A} [f_{y1} \Delta u_{y1} + f_{y2} \Delta u_{y2}]$$
(26)

Hence

1

$$G_{total} = G_1 + G_{II}$$
(27)

RESULTS AND DISCUSSION

The presence of circumferential crack between the ceramic layer and the bond coat layer as shown in Fig. 1 was analyzed using A Rayleigh-Ritz method and finite element method. The general code ABAQUS was used for finite element analysis. The energy release rate G was estimated by using the virtual crack extension method.

Finite element method was used in order to determine the J-integral value and the energy release rate. Virtual crack extension method was used to evaluate the energy release rate for both Mode I and Mode II crack growth. For stepped-disk specimen, the disk radius is 12.7 mm, the bond coat layer thickness is 0.04826 mm, the TBC thickness is 0.127 mm, and the uncoated Nickel based superalloy had a thickness of 3.175 mm. These dimensions are shown in Fig. 4. The properties of these three layers are given in Table 1. We analyzed a model case where the specimen was plasma sprayed in air with a thin zirconia-yttria

 $(ZrO_2 - 8wt\%Y_2O_3)$ layer on a nickel-chromium-aluminum-zirconium bond coat, as in Ref. 1. The specimen was cooled down from a processing temperature of 1000 $^{\circ}C$ to a temperature of 0 $^{\circ}C$. Half model for stepped-disk specimen was used since the specimen is axisymmetric. Eight node isoparametric element type was used; the total number of element was 1380. Along the longitudinal axis, the nodes can move in the axial direction only. The mesh was very fine close to the crack tip, and to make sure that the mesh is fine enough, another model with the same number of element but has one tenth of element size near to the crack tip, the difference in the results was less than 1%. Details of the finite element mesh near the crack tip is shown in Fig. 5.

Figure 6 shows the variation of maximum deflection versus pressure ratio for a delamination radius of 20 times the TBC thickness. For zero pressure load the deflection has a value greater than zero due to buckling. It is clear that the use of temperature dependent properties in contrast to constant properties provide significantly different values of maximum deflection. The finite element results compare to numerical results correlate very well. The variation of the maximum deflection versus pressure ratio is non-linear compare to linear variation as in the case of no geometrical nonlinearity. Using temperature dependent properties gives a 20-30% higher maximum deflection and energy release rate as seen in Figs. 6 and 7. The variation of J and G_t versus pressure ratio from finite element method and virtual crack extension method (VCEM) are shown in Fig. 7. Comparison between the energy release rate evaluated by energy method and by finite element method (J-integral) is shown in Fig. 8, finite element method and numerical method are very good agreement.

Figure 9 shows the variation of G_{I} and G_{II} versus pressure ratio for a delamination radius of twenty times the TBC thickness. This figure shows good agreement between finite element method results based on VCEM and numerical results. Mode I is dominant in this case. Figure 10 is same as Fig. 9, but temperature dependent properties were used. In Fig. 10, we observe that Mode II energy release rate component is much smaller compared with the mode I component for both numerical and VCEM analyses. In either case, results match well. Figure 11 shows the variation of J-integral delamination radius from numerical analysis. From this figure one can obtain the conditions when the crack will propagate. From literature the critical energy release rate is varied between 100-300 $J.m/m^2$ for this system configuration. For a value of 300 J.m/m^2 , and a delamination radius of four times the TBC thickness, the crack will propagate at induced pressure value equivalent to 500 atmosphere (\approx 50 MPa). This value is easy to develop due to volume increase because of oxide layer growth. Also, based on finite element analysis conducted by the authors for this system with a wavy interface, the results shows that an axial stress of 250 MPa can be developed for a sine wave interface with amplitude of 2.4 µm and wave length of 127 µm. These values are practical for this system (Newaz, et al., 1996).

CONCLUSIONS

Effect of interfacial microcracks were investigated using fracture mechanics approach under thermo-mechanical loading. Thermal load was used in conjunction with internal surface pressure due to oxide scale buildup to analyze interfacial cracks. The use of temperature dependent properties in contrast to constant properties provide significantly different values of J-integral and G values. Stepped-disk type specimen with a central crack has mixed mode conditions under pure thermo-mechanical loading. The total energy release rate evaluated by the virtual crack extension method and the J-integral value evaluated directly by ABAQUS agree quite well. The maximum deflection and total energy release rate evaluated by finite element method and energy method are in very good agreement. For a

TBC toughness value of 300 J.m/m 2 , and a delamination radius of four times the TBC thickness, the crack will propagate at induced pressure value equivalent to 500 atmosphere

(\approx 50 MPa). The results clearly indicate that small internal pressure due to oxidation induced volume change may create the necessary conditions for crack growh during thermal cycling.

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TABLE 1

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Material	Young's	Poisson's ratio	Coefficient of
	modulus (GPa)		thermal
`			expansion
			(⁰ C ⁻¹)
Substrate	175.8	0.25	13.01×10^{-6}
	150.4	0.2566	13.71 X10
	94.1	0.3224	15.36 x10 ⁻⁰
			19.52×10^{-6}
Bond coat	137.9	0.27	15.16×10^{-6}
	121.4	0.27	15.10 X10
	93.8	0.27	15.37×10^{-0}
			17.48×10^{-6}
TBC	27.6	0.25	10.01×10^{-6}
	6.9	0.25	-6
	1.84	0.25	11.01 x10
			12.41×10^{-6}

Material properties at 22 & 566 & 1149 $^{
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Figure 1. Circumferential crack between the ceramic layer and the bond coat layer in a stepped-disk specimen.



Figure 2. Notation and parameters used for J-integral.

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Figure 3a. Interfacial crack between bond coat and TBC.



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Figure 3b. Forces and displacement illustrations for a crack.



Figure 4. Dimension of TBC layer in relation to bond coat and superalloy substrate.



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Figure 5. Detailes of near crack-tip finite element mesh



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Figure 6. Variation of maxmium deflection versus pressure ratio, for both constant and temperature dependent properties, a=20h.



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Figure 7. Variation of the J & G versus pressure ratio, for both constant and temperature dependent properties, a=20h.



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Figure 8. Variation of J & G versus pressure ratio, numerical and finite element metod are used, for both constant and temperature dependent proprties, a=20h.



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Figure 9. Variation of G_I & G_{II} versus pressure ratio, numerical and finite element methods are used, constant properties, a=20h.



Figure 10. Variation of $G_1 \& G_{11}$ versus pressure ratio, numerical and finite element method are used, temperature dependent properties, a=20h.



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Figure 11. Variation of G versus delimination radius at different pressure ratio, numerical and temperature dependent properties are used.

Submitted to the J. Materials Science, U.K. October, 1996. / Accepted July, 1997, CHRONOLOGICAL EVALUATION OF INTERFACIAL DAMAGE IN TBC DUE TO THERMAL CYCLING

Z. A. Chaudhury¹, G. M. Newaz¹, S. Q. Nusier¹, T. Ahmed² and R. L. Thomas²
 Mechanical Engineering Department¹, Institute of Materials Research²
 Wayne State University, Detroit, MI 48202, USA

and

K. P. Wright General Electric Aircraft Engines Evendale, OH 45215, USA

ABSTRACT

A two layer Electron Beam-Physical Vapor Deposited (EV-PVD) Thermal Barrier Coating (TBC) on a single crystal superalloy (Rene' N5) substrate was characterized prior to and after thermal cycling at 2, 18, 25, 44, 50, 75, 100, 110, 150 and 175 cycles in between 200 C-1177 C. Optical microscopy, scanning electron microscopy and thermal wave imaging techniques were used to characterize the interfacial damage. Pt-Al was used as bond coat and 8 wt.% YSZ was used as outer top layer. Interfacial cracking was observed even at 2 thermal cycles. Thermally Grown Oxide (TGO) layer increased with the number of thermal cycles. After numerous cycles over 100, interfacial separation was observed to be higher at the middle than at the edges of the sample. This observation is consistent with buckling induced delamination - a possible mechanism for spallation.

CHRONOLOGICAL EVALUATION OF INTERFACIAL DAMAGE IN TBC DUE TO THERMAL CYCLING

Z. A. Chaudhury¹, G. M. Newaz¹, S. Q. Nusier¹, T. Ahmed² and R. L. Thomas²
 Mechanical Engineering Department¹, Institute of Materials Research²
 Wayne State University, Detroit, MI 48202, USA

and

K. P. Wright General Electric Aircraft Engines Evendale, OH 45215, USA

1. Introduction

Different turbine engine components are using thermal barrier coatings(TBCs) in order to increase the life of the metal parts and to improve the operating temperature. TBCs offer a number of benefits including up to 145 C metal temperature reduction, significant fuel savings and improved durability [1, 2]. Other advantages and improvements of TBCs on the turbine blades are to increase the turbine thermal efficiency due to a higher stator outlet temperature, increase the compressor efficiency due to a reduced air flow for the turbine cooling, longer service life of the metallic substrate due to a decreased thermal fatigue load. TBCs consist of a thermally insulating zirconia ceramic overlayer applied over a metallic bond coat which protects the substrate from oxidation and hot corrosion during high temperature operations. Several coating methods are available. Of them Plasma Spray and Electron-Beam Physical Vapor Deposition (EB-PVD) processes are very popular for their various advantages. EB-PVD has been successfully used to coat turbine airfoils [3]. EB-PVD ceramic structure is highly columnar with the columns aligned perpendicular to the metalceramic interface. Due to columnar microstructure in EB-PVD system, the coatings become strain tolerant. The reason for this is that the coatings can accommodate the applied thermal strain by movement between the columnar growth structures [2]. In EB-PVD technique a small piece of ceramic material such as 8 wt.% YSZ is evaporated by an electron beam in a vacuum chamber. The ceramic vapor cloud condenses on the surfaces of the component, which is rotated and controlled in the vapor cloud to form the coating. Power to evaporate the ceramic coating material is provided by a high-energy electron beam gun. Feeders, electron beam gun, temperature and gas should be controlled very carefully.

Calculations indicate that 0.25 mm thick layer of zirconia can reduce metal temperature by as much as 170 C depending on local heat flux [1, 4]. This relative temperature reduction can provide significant benefits [1, 2] in the form of either component durability extension or system performance improvement. The most widely used TBCs are based on zirconia (zirconium dioxide), which shows thermal expansion characteristic similar to superalloys. Zirconia offers a good compromise with a low thermal conductivity about (2 W/mK like 3 to 7% of superalloy) and a high thermal expansion coefficient (~ 10×10^{-6} K⁻¹) [5]. Yttria Stabilized Zirconia (YSZ) coatings constitute the current state of the art of TBCs [6].

Bond coat compositions started from early Ni-Cr and Ni-Al compositions to the presently used MCrAlX (M=Ni or Cr or both, X=Hf, Zr or Al) and Pt-Al compositions. Pt-Al diffusion coating has been developed due to demands on coatings for gas turbine engines, that must operate at significantly higher temperatures than required previously. Pt-Al is also cheaper in the long run process. Oxidation behavior of platinum-modified aluminide coating and platinum -aluminum alloys has been reported by other investigators [7, 8].

The function of the bond coat in a TBC system is to provide good adhesion between the metal substrate and the ceramic top coat while providing good oxidation protection to the underlying substrate alloy. During thermal exposure, this bond coat oxidizes and forms an aluminum oxide layer between the ceramic and the bond coat. Oxidation plays a dominant role in the failure of graded thermal barrier coatings as confirmed by many researchers [9-12]. Oxidation of the bond coat has been proposed [13] as a life limiting factor of TBCs. Numerous studies have shown that oxidation of bond coat can significantly affect spalling [14-17].

Depending on the temperature range to which thermal barrier coatings are exposed, different types of failure modes have been observed. Bond coat oxidation and damage initiation is common for high temperature applications [18-20]. On the other hand for low temperature applications thermal cyclic stresses which occur during engine operation is believed to be responsible for coating delamination and spallation [21]. For long term successful use of the turbine components, the durability of the TBC must be maintained. Due to improvements in processing condition and coating materials,

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significant advances in coating durability have been realized [22-28]. Stress calculations have indicated that high rates of heating and cooling has a more destructive influence on ceramic coating life than isothermal exposure at temperature [29]. Substrate temperature is important in TBC behavior. Thermal cycle life of TBC decrease dramatically due to higher substrate temperature [30-31]. Compressive stresses are also believed to be responsible for TBC failure which occur in the ceramic layer during cooling [14, 29]. The mechanism involves buckling of TBC due to compressive residual stress which results in spallation. These stresses can be attributed to the thermal expansion mismatch between the ceramic top coat and the metal bond coat[32-35]. Extensive research work on TBCs have been reported in the literature [36-46]. The basic causes of TBC failure and the effect of TGO on delamination is still not clear. As a part of our continued research program on an investigation with focus on damage accumulation mechanisms in thermal barrier coated single crystal substrate [47-49], a number of thermal cycling tests have been carried out, evaluated and analyzed to determine the causes of coating failure and delamination. In this article, we present a chronological sequence of damage and oxidation evolution at the bond coat and TBC interface.

2. Experimental Procedure

The TBC specimens were 25.4 mm diameter by 3.17 mm thick button samples of nickel base superalloys Rene' N5. There are two coats. One is the top coat which is the EB-PVD thermally insulting outer ceramic layer and the other is the bond coat which is a

diffusion aluminide Pt-Al alloy. This bond coat protects the alloy substrate from oxidation and bonds very well to both the alloy substrate and the outer EB-PVD thermally insulating layer. The outer layer TBC has been the 8 wt.% YSZ. The thickness of the outer top coat is 0.127 mm and that of bond coat is 0.0482 mm. The outer ceramic top coat is used for its chemical and thermal stability as well as low thermal conductivity and relatively high coefficient of thermal expansion. Several researchers have worked on this material systems between 150 C 1200 C. A number of coated specimens were placed in a muffle-type rapid heating/cooling furnace and thermally cycled to 2, 18, 25, 42, 50, 75, 100, 110, 150, and 175 cycles in the range of (200 C- 1177 C). The holding time was 45 minutes at the peak temperature in each cycle. The rate of heating was 9 minutes to reach to the peak temperature and the rate of cooling was 10 minutes to reach to the lower temperature of the cycles. Figure 1 shows the temperature profile of the thermal cycle tests.

A number of samples were taken out after a certain number of thermal cycles. These samples did not fail. The thermally cycled samples were potted by using cold mounting epoxide resin. After curing for one day, the specimens were cut by using a Buheler Isomet 2000 diamond cutter. The samples were then grinded and polished by using normal metallurgical procedures. The polished samples were observed under Olympus BX 60 optical microscope. Scanning Electron Microscopy was carried by using a Hitachi 2000 SEM on the polished samples to observe interfacial damage/crack propagation at the ceramic top coat and bond coat interface and morphology of the thermally grown oxide (TGO) layer at higher magnification. Thermal wave images were taken on the surface of

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the thermally cycled and untested samples in order to identify any damage at the ceramic top coat and bond coat interface.

3. Results and Discussions

3.1 Thermal Cycling of TBC samples

Figure 2 shows the photomicrograph of a thermal barrier coated sample without any thermal cycling. No interfacial cracking and oxidation product (TGO) were observed at the bond coat/top coat (TBC) interface. Thermally cycled TBC samples in air leads to the formation of a reaction product layer and interfacial cracking/damage at the bond coat/top coat interface. This oxidation product (TGO) is Al₂O₃ and has been confirmed by many researchers [47, 48, 51, 52]. Interdiffusion of bond coat and substrate elements at elevated temperature does occur [53] and their effect on TBC failure is still not clear. Migration of aluminum from bond coat into the substrate can change the oxidation behavior of the bond coat and sometimes may be responsible for the formation of less adherent oxide species. A few number of thermal cycles leads to the formation of interfacial cracking at the bond coat/top coat interface and with increasing the number of cycles leads to the formation of both interfacial cracking and TGO layer. Figures 3 (a) and 3 (b) show the photomicrographs of the interfacial cracking at the bond coat/top coat interface taken after 2 and 18 thermal cycles. TGO layer just started growing at 18 thermal cycles. The length of the interfacial crack was typically 600 µm and the thickness of the crack was 3 µm. The TGO layer starts growing with increasing the number of thermal

cycles. Figures 3(c)-(d), 4 (a)-(c), 5 and 6(a)-6(b) show the chronological evaluation of interfacial damage in TBC due to thermal cycling at 25, 42, 50, 75, 100, 110 and 175 cycles. The thickness of the TGO layer grows with increasing the number of cycles. Figure 4 (c) shows separation through the TGO layer. In figure 5, interfacial cracking at the bond coat/top coat interface are increased and TGO layer is quite visible through the interfacial cracking. Interfacial cracking is showing a sinusoidal nature. In Figures 6 (a)-6 (b), complete separation through TGO layer is observed. This TGO layer/separation is very high at the center and small at the edges of the TBC sample. The thickness of this separation at the center is roughly double than at the edges which is $\sim 85.5 \ \mu m$ at the center and \sim 42.9 μm at the edges. Figure 7 shows the schematic cross-section and photomicrographs of TBC specimen showing interfacial separation through TGO layer after 150 thermal cycles. The TGO layer is thicker at the middle and thinner at the edges of the specimen. A large interfacial delamination crack is needed for buckling induced spallation to occur. Current understanding [9, 12] and from the Figures 5, 6 (a)-6 (b), and 7 it is now clear that buckling induced delamination is a possible mechanism for spallation.

3.2 Scanning Electron Microscopy (SEM)

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Scanning Electron Microscopy is a very good tool for materials characterization at higher magnifications. Sample preparation for SEM is simple. SEM was done on all the thermally cycled samples. Figures 8(a)-(c) show the high magnification SEM micrographs taken from the specimens thermally cycled at 25, 50 and 150 cycles. At 25 thermal cycles, separation through TGO layer is quite prominent as shown in the micrograph 8(a). In

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micrograph 8(b), voids and microcracks are seen and TBC seems to be spalling out. Figure 8 (c) shows the microcrack and big separation. Interfacial damage is very large in this situation.

3.3 Nondestructive Evaluation Using Thermal Wave Imaging (TWI)

Microscopy is a destructive technique and time consuming. TWI on the other hand can provide a good assessment of damage due to thermal loading without any destruction of the samples. It is clean, cheap and much faster than microscopy.

The thermal wave imaging method is based on the idea in which a time-dependent heat source at the surface of an object launches waves of heat into the object in the form of heat diffusion, called thermal waves. The thermal waves are scattered from subsurface defects or anomalies in much the same way that sound waves reflect from such defects and, upon returning to the surface of the object, modify the temperature of the surface map. Figure 9 shows the schematic experimental setup for thermal wave imaging[54]. In the usual configuration, high power flash lamps are used to pulse-heat the surface of the object under inspection. This causes a plane thermal wave pulse to propagate into the material from the heated surface. As this pulse encounters subsurface material defects, each defect scatters a fraction of the pulse back towards the surface. When these scattered pulses or thermal waves arrive back at the surface, they modify the time dependent temperature distribution on the surface, with signal from defects at different depths affecting the surface temperature at different times. During the process, the evolving surface temperature distribution is imaged by an infrared video camera as function of time. Through the use of fast image processing hardware and software, the system's computer memory stores a sequence of gated images corresponding to the various times after the flash heating. The result is a series of thermal wave images corresponding to various depths beneath the surface. The time after the onset of the flash heating at which defects from various depths beneath the surface can be observed depends on the depth and the thermal diffusivity of the specimen under inspection.

Figure 10 (a) shows the thermal wave image of four samples at 0, 25, 50 and 100 thermal cycling respectively from left to right. In this image the color map representation is as follows: blue is low thermal wave signal amplitude, followed by green and yellow indicating increasing thermal wave signal amplitude respectively and red being the higher thermal wave signal amplitude. The corresponding surface temperature profile plots shown on the right hand side of the figure represents the relative amplitude plotted as function of position or distance across the center of the samples. The plot indicates that the thermal wave signal amplitude increases 100% from 0 thermal cycling to 25 thermal cycling, while the change in the thermal wave signal amplitude from 25 thermal cycling to 50 thermal cycling is only 10% for each case. Figure 10 (b) shows the thermal wave image of a second set of samples at 0, 150 and 175 thermal cycling respectively from left to right. The color map used is identical to the one in figure 10(a). The corresponding surface temperature profile shows that the thermal wave signal amplitude increases 150% from 0 thermal cycling to 150 thermal cycling, however, the thermal wave signal amplitudes of the 150 thermal cycling and 175 thermal cycling samples are identical. As the number of thermal cycles increases, the damage at the bond coat/top coat interface are

also increased. The damage thickness may also change depending on the number of thermal cycles. This damage thickness may be responsible for the thermal wave signal amplitude to vary.

4. Conclusions

Based on our present investigation, the following conclusions may be drawn:

(1). Microcracks may occur early due to thermal cycles. A number of microcracks were observed only after 2 cycles (200 C-1177 C) in the bond coat near the TBC/bond coat interface.

(2). The TGO layer continues to grow with higher the number of thermal cycles. However, there is a substantial interaction of TGO layer and damage at higher number of cycles.

(3). There appears to be a transition from low scale distribution damage (microcracks that are separate) to more extensive interlinked damage (microcracks that have connected) around 100 cycles.

(4). Interfacial separation between bond coat and TBC increases with increasing the number of thermal cycles. This separation is higher at the center and lower at edges of the sample. The separation has been estimated to be 85 μ m for 175 thermal cycles. This separation is twice the separation measured at the edges of the sample.

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(5). Based on current evidence and from our previous investigation [50], it can be concluded that buckling induced delamination is a possible mechanism for spallation.

(6). Thermal wave imaging technique is potential NDE tool to assess the degradation in TBC system.

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FIGURE CAPTIONS

- 1. Figure 1 shows the temperature profile of the thermal cycle tests conducted in the present investigation.
- 2. Figure 2 shows an optical micrograph of an untested thermal barrier coated sample. Thermally Grown Oxide (TGO) layer and/or interfacial separation were absent at the bond coat/top coat (TBC) interface.
- 3. Figures 3.(a) and (b) show the photomicrographs of interfacial cracking at the bond coat/top coat interface taken after 2 and 18 thermal cycles. TGO layer started growing at 18 cycles. Figures 3 (c) and (d) show the chronological evaluation of interfacial damage in TBC due to thermal cycling at 25 and 42 cycles.
- 4. Figures 4(a), (b) and (c) are the optical micrographs showing the chronological evaluation of interfacial damage in TBC due to thermal cycling at 50 and 75 and 100 cycles. Separation through TGO layer is quite visible.
- 5. Figure 5 shows the photomicrograph of bond coat cracking in between bond coat and TBC after thermal cycled at 110 cycles. TGO layer is visible through the bond coat cracking. This bond coat cracking is sinusoidal nature.
- 6. Figure 6 (a) and (b) are the optical micrographs showing complete separation through the TGO layer after thermal cycled at 175 cycles. Figure 6 (a) shows the separation at the edge of the specimen and Figure 6(b) shows the separation at the center of the specimen.

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- 7. Figure 7 show the schematic and photomicrographs of TBC specimen showing interfacial separation through TGO layer after thermal cycled at 150 cycles.The photomicrographs were taken from two edges and center of the specimen.
- 8. Figure 8 show the scanning electron micrographs taken from the polished TBC specimens after thermal cycled at 25, 50 and 150 cycles. TGO layer, microcracks, voids and separation through TGO layer are observed in those specimens.
- 9. Figure 9 shows a schematic experimental setup for thermal wave imaging.
- 10. Figure 10 (a) shows the thermal wave image of four samples at 0, 25, 50 and 100 thermal cycles and their corresponding surface temperature profile plots respectively. Figure 10 (b) shows the thermal wave image of other set of samples at 0, 150 and 175 cycles and their corresponding surface temperature profile plots.











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Fig.4



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Comparison of thermal wave images at various thermal cycling and corresponding surface temperature profile across the specimens





Distance (pixel coordinate)





Experimental setup for thermal wave imaging.

