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HANDBOOK FOR RECOMMENDED MATERIAL REMOVAL PROCESSES FOR ADVANCED CERAMIC TEST SPECIMENS AND COMPONENTS

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FOREWORD

This handbook is published by the National Aeronautics and Space Administration (NASA) as a guidance document that provides engineering information; lessons learned; possible options to address technical issues; classification of similar items, materials, or processes; interpretative direction and techniques; and any other type of guidance information that may help the Government or its contractors in the design, construction, selection, management, support, or operation of systems, products, processes, or services.

This handbook is approved for use by NASA Headquarters and NASA Centers, including Component Facilities.

This handbook establishes guidelines and recommendations for machining of advanced ceramics. Machining of advanced ceramics is often necessary to achieve certain design requirements such as dimensional (tolerance) requirements, geometric shape, functional fit, and surface finish. However, surface grinding can cause a significant decrease in the strength of advanced ceramics due to the introduction of surface flaws. The magnitude of the loss in strength is determined by the grinding conditions and the response of the material. The effect on strength of varying a single grinding parameter or several grinding parameters can be measured and assessed; however, doing so can be both time consuming and expensive depending on geometry, application, and material. Often grinding procedures have been developed by experienced users for particular components or applications, but these procedures have not been compiled or presented in a generalized manner accessible to inexperienced users. Therefore, a need exists to compile and/or develop a set of geometry-based grinding procedures that inexperienced users of advanced ceramics can apply as a starting point in specification writing and fabrication of ceramic test specimens and components.

Requests for information, corrections, or additions to this handbook should be submitted via "Feedback" in the NASA Technical Standard System at http://standards.nasa.gov.

Original Signed By

____________________________________ ___11/19/2007___

Michael G. Ryschkewitsch
NASA Chief Engineer

Approval Date

TABLE OF CONTENTS

SECTION		PAGE
DOCUMENT HISTORY LOG		2
FOREWORD		3
TABLE OF CONTENTS		4
LIST OF FIGURES		5
LIST OF TABLES		5
1.	**SCOPE**	6
1.1	Purpose	6
1.2	Applicability	6
2.	**APPLICABLE DOCUMENTS**	7
3.	**ACRONYMS AND DEFINITIONS**	7
3.1	Acronyms	7
3.2	Definitions	7
4.	**GUIDANCE**	8
4.1	Significance and Use	8
4.2	Interferences	9
4.3	Apparatus	9
4.4	Precautionary Statement	10
4.5	Recommended Procedures for Specific Configurations	10
4.6	Post-Machining Treatments	14
5.	**ADDITIONAL GUIDANCE**	14
5.1	Reference Documents	14
5.2	Intended Use	15
5.3	Key Word Listing	15
APPENDIX A	**RECOMMENDED POLISHING SPECIFICATIONS FOR CERAMIC WINDOWS**	16

LIST OF FIGURES

Figure	Title	Page
1	Horizontal-Spindle Surface Grinding for Machining Prismatic Uniaxial Flexure Bars	10
2	Vertical-Spindle Surface (Blanchard) Grinding and Polishing for Machining Biaxial Flexure Disks	11
3	Outside Diameter Cylindrical Grinding for Machining Cyclindrical Tension/Compression Rods	13

LIST OF TABLES

Table	Title	Page
1	Scratch/Dig Specification Details	20

Handbook for Recommended Material Removal Processes for Advanced Ceramic Test Specimens and Components

1. SCOPE

1.1 Purpose

This handbook covers recommended material removal processes (i.e., machining or grinding) for advanced ceramics. It is applicable to both test specimens and components, and hereafter they will be referred to as “specimens.” This handbook is not intended to replace or supercede customary (e.g., internally accepted or proprietary) or application-matched machining/grinding practices. Instead, it is intended to provide recommended material removal procedures developed from experience and testing, and thereby ensure consistent test specimen and component performance. Geometries addressed in this standard practice include prismatic sections, flat plates (disks and square plates), and cylindrical rods. Grinding parameters, including diamond (abrasive)-grit size and material removal rates, are addressed in addition to cutting fluid type and conditions. Appendix A provides a specific application example, namely: Recommended Polishing Specifications for Ceramic Windows.

Fabrication of test specimens and components can introduce dimensional variations, subsurface damage, and residual stresses which may have pronounced effects on measured mechanical properties and behavior. Because universal or standardized procedures for surface preparation do not exist, guidance on specimen preparation is useful to ensure that such variations are minimized in determining material properties such as ultimate strength. The procedures described in this handbook address some of the factors responsible for machining effects. It should be understood that final machining steps may or may not negate machining damage introduced during the initial steps. Therefore, measures like surface roughness alone of the specimen may not be adequate for determining ultimate strengths of advanced ceramics. Specimen fabrication processes should be controlled and reported.

1.2 Applicability

This handbook may be referenced in contract, program, and other Agency documents for guidance. Individual portions of this handbook may be tailored (i.e., modified or deleted) by contract or program specifications to meet specific program/project needs and constraints. Tailoring must be formally documented and approved as part of program/project requirements.

This practice may involve hazardous materials, operations, and equipment. This test method does not purport to address the safety problems associated with its use. It is the responsibility of the user of this practice to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

This practice is intended primarily for use with advanced ceramics and optical materials that "macroscopically exhibit" isotropic, homogeneous, continuous behavior. While this practice is intended for use on monolithic advanced ceramics and optical materials, certain whisker- or particle-reinforced composite ceramics as well as certain discontinuous fiber-reinforced composite ceramics may also meet these macroscopic behavior assumptions. Generally, continuous fiber ceramic composites (CFCCs) do not macroscopically exhibit isotropic, homogeneous, continuous behavior; and application of this practice may not be appropriate.

Values expressed in this handbook are in accordance with the International System of Units (SI) and American Society for Testing and Materials (ASTM) SI10.

2. APPLICABLE DOCUMENTS

ASTM C 1145	Terminology on Advanced Ceramics

3. ACRONYMS AND DEFINITIONS

3.1 Acronyms

ASTM	American Society for Testing and Materials
CFCCs	Continuous Fiber Ceramic Composites
CNC	Computer Numerical Control
min	minute
NASA	National Aeronautics and Space Administration
OD	outer diameter
SI	International System of Units

3.2 Definitions

The following definitions of applicable terms are taken from ASTM C 1145, Terminology on Advanced Ceramics:

Advanced Ceramic: A highly engineered, high-performance, predominately non-metallic, inorganic, ceramic material having specific functional attributes.

Extraneous Flaws: Strength-controlling flaws observed in some fraction of test specimens that cannot be present in the component being designed. An example is machining flaws in ground bend specimens that will not be present in as-sintered components of the same material.

Fractography: The analysis and characterization of patterns generated on the fracture surface of a test specimen. Fractography can be used to determine the nature and location of the critical fracture origin. Such fractures can cause catastrophic failure in an advanced ceramic test specimen or component.

Intrinsic Flaws: Strength controlling flaws observed in some fraction of test specimens that can be present in the component being designed. Examples are pores and agglomerations which are formed during processing and consolidation of the advanced ceramic.

Machining Damage: As used in fractography, chips and surface subsurface microcracks, striations, and scratches created during the machining process.

Slow Crack Growth: Sub-critical crack growth (extension) that may result from, but is not restricted to, such mechanisms as environmentally assisted stress corrosion or diffusive crack growth.

Note: In the ceramics literature, "slow crack growth curve" is often called a "static fatigue" curve.

4. GUIDANCE

4.1 Significance and Use

4.1.1 Scope

This practice may be used for material development, material comparison, quality assurance, characterization, and design data generation.

4.1.2 Extraneous Flaws

Generally, strength distributions of ceramics are probabilistic and can be described by a weakest link failure theory. These strength distributions can be related to distributions of both extraneous and intrinsic flaw distributions. In determining the intrinsic strength distribution of an advanced ceramic, it is important to limit the effect of extraneous flaws, particularly those introduced by machining, grinding, lapping, and polishing the test specimens.

4.1.3 Application-matched Machining

In cases where customary or application-matched machining or grinding procedures have not been developed, a consistent, recommended machining or grinding practice can be useful as a starting point for developing such procedures.

4.2 Interferences

4.2.1 Fabrication Effects

Fabrication of specimens can introduce dimensional variations and/or damage which may have pronounced effects on measured mechanical properties and behavior. Machining effects introduced during test specimen preparation can interfere in determining the ultimate strength of pristine materials. Surface preparation can also lead to the introduction of residual stresses. Although universal or standardized procedures for surface preparation do not exist, the procedures described in this practice attempt to address some of the factors responsible for machining effects. It should be understood that final machining steps may or may not negate machining damage introduced during the initial machining. Therefore, although surface roughness in the gage section of the test specimen may or may not be critical for determining ultimate strengths of advanced ceramics, test specimen fabrication history may play an important role in the measured strength distributions and should be reported.

4.2.2 As-Processed Surfaces

In addition, the nature of fabrication used for certain advanced ceramics (e.g., pressureless sintering, hot pressing) may require the testing of specimens with gage sections in the as-processed condition. Therefore, it may not be possible or desired/required to machine some test specimen surfaces not directly in contact with test fixture components. For very rough or wavy as-processed surfaces, eccentricities in the stress state due to non-symmetric cross sections as well as variations in the cross-sectional dimensions may also interfere with the stress or strength determination.

4.2.3 Tolerances

Finally, close geometric tolerances, particularly in regard to flatness, concentricity, and cylindricity of test specimen surfaces or geometric entities in contact with the test fixture components are critical requirements for successful mechanical tests.

4.3 Apparatus

4.3.1 Machines for Material Removal Processes

Use only suitable machines for material removal processes applied to advanced ceramics (e.g., diamond-grit cutting and grinding, electro-discharge machines, abrasive water jets, etc.). No generally accepted minimum requirements for such machines have been developed.

4.4 Precautionary Statement

4.4.1 Dust as a Health Hazard

Grinding and cutting advanced ceramics often create fine particles which may be a health hazard. Materials containing whiskers, small fibers, or silica particles may also cause health hazards when compression tested. For such materials, the operator is advised to consult the material safety data sheet for guidance prior to testing. Suitable ventilation or masks may be warranted.

4.5 Recommended Procedures for Specific Configurations

4.5.1 Prismatic Uniaxial Flexure Bars (e.g., as used in ASTM Test Method C 1161)

4.5.1.1 Grinding Procedure

All grinding should be done with an ample supply of appropriate filtered coolant to keep the workpiece and wheel constantly flooded and particles flushed. Grinding in at least two stages, ranging from coarse to fine rates of material removal, is recommended. All machining is done in the surface grinding mode, parallel to the specimen's long axis, as shown in figure 1. Do not use Blanchard or rotary grinding. (See figure 2.)

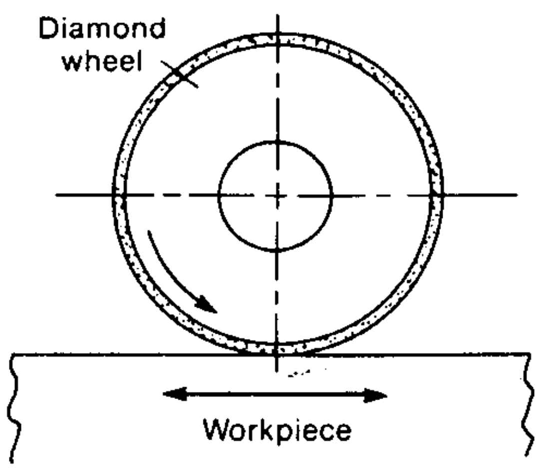

Figure 1—Horizontal-Spindle Surface Grinding for Machining Prismatic Uniaxial Flexure Bars

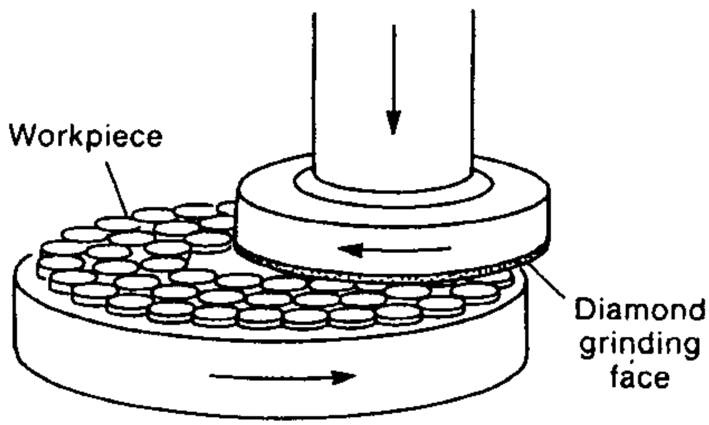

Figure 2—Vertical-Spindle Surface (Blanchard) Grinding and Polishing for Machining Biaxial Flexure Disks

4.5.1.2 Stock Removal Rate

The stock-removal rate should not exceed 0.03 mm per pass to the last 0.06 mm per face. Final (and intermediate) finishing should be performed using a diamond wheel that is between 320 and 500 grit. Remove 0.06 mm per face during the finishing phase, and at a rate of not more than 0.002 mm per pass. Remove approximately the same amount of equal stock from opposite faces.

4.5.1.3 Low Toughness Materials

Materials with low fracture toughness and a greater susceptibility to grinding damage may require finer grinding wheels at very low removal rates.

4.5.1.4 Chamfers

Chamfer the four long edges of each specimen uniformly to 45°, a distance of 0.12 ± 0.03 mm, or they can be rounded with a radius of 0.15 ± 0.05 mm. Edge finishing is comparable to that applied to the specimen surfaces. In particular, ensure that the direction of machining is parallel to the test specimen long axis. If chamfers are larger than the tolerance allows, then make corrections to the stress calculations. As an alternative, if a test specimen can be prepared with an edge that is free of machining damage, then a chamfer is not required.

4.5.2 Flat Biaxial Flexure Disks (e.g., as used in ASTM Test Method C 1499.)

4.5.2.1 Grinding Procedure

Perform all grinding or cutting with an ample supply of appropriate filtered coolant to keep the specimen and grinding wheel constantly flooded and particles flushed. Grinding can be done in two stages, ranging from coarse to fine rates of material removal. All cutting can be done in one stage appropriate for the depth of cut. (See figure 2.)

4.5.2.2 Stock Removal Rate

Ensure the stock-removal rate does not exceed 0.03 mm per pass to the last 0.06 mm of material removed. For final finishing, use diamond tools between 320 and 500 grit. Remove no less than 0.06 mm during the final finishing stage, and at a rate less than 0.002 mm per pass. Remove equal stock from opposite faces.

Note: For alpha silicon carbide (and some aluminum oxides and silicon nitrides), annealing at ~1200 °C for ~2 hours was sufficient to "heal" the grinding damage induced by the procedure in section 4.5.2.2 without otherwise altering the materials strength. However, note that annealing can significantly alter materials properties; and specific procedures need to be developed for each material.

4.5.2.3 Heat Treatment

Grinding may be followed by either heat treatment or lapping, as deemed appropriate. See Appendix A for additional details on polishing ceramic windows. The purpose of such treatments is to eliminate damage introduced during machining and thereby sample only intrinsic flaws. If the component will have such machining damage, then eliminate this step so that similitude exists between the test specimen and the component.

Note: For lapping of alpha silicon carbide, the following procedure was successful in elimination of machining damage induced by uniaxial grinding: successive lapping with 15, 9, and 6 µm diamond pastes for ~30, ~25, and ~15 min, respectively. For tungsten carbide, successive machine lapping with 15 µm, and 6 µm diamond pastes for 30 min each was sufficient. Specific procedures need to be developed for other materials.

4.5.2.4 Orientation Marks

To aid in post failure fractographic examination, it is recommended that the orientation of the grinding direction be marked on the specimens. This marking can be accomplished with an indelible marker.

4.5.3 Cylindrical Tension/Compression Rods (e.g., as used in ASTM Test Methods C 1273 and C 1424.)

4.5.3.1 Grinding Procedure

Perform all grinding or cutting with an ample supply of appropriate filtered coolant to keep the workpiece and grinding wheel constantly flooded and particles flushed. Grinding can be done in two or more stages, ranging from coarse to fine rate of material removal. All cutting can be done in one stage appropriate for the depth of cut. (See figure 3.)

4.5.3.2 Stock Removal Rate

Ensure stock removal rate does not exceed 0.03 mm per pass up to the last 0.06 mm of material removed using diamond tools that have between 320 and 500 (or 600) grit. No less than 0.06 mm should be removed during the final finishing phase, and at a rate not more than 0.002 mm per pass. Remove equal stock from each surface where applicable.

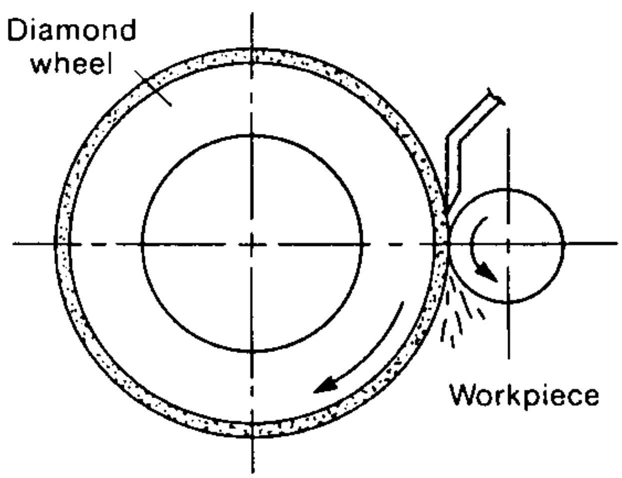

Figure 3—Outside Diameter Cylindrical Grinding for Machining Cylindrical Tension/Compression Rods

4.5.3.3 Grinding Direction

Because of the axial symmetry of the contoured compressive test specimen, fabrication of the test specimens is generally conducted on a lathe-type apparatus. In many instances for tensile test specimens, the bulk of the material is removed in a circumferential grinding operation; and a final longitudinal grinding operation is then performed in the gage section. Such a final longitudinal grinding operation is not necessary for compressive test specimens because of the bulk-related (i.e., not weakest link) compressive strength mechanism.

4.5.3.4 Specimen Mounting

Generally, computer numerical control (CNC) fabrication methods are necessary to obtain consistent test specimens with the proper dimensions within the required tolerances. A necessary condition for this consistency is the complete fabrication of the test specimen without removing it from the grinding apparatus, thereby avoiding introduction of unacceptable tolerances into the finished test specimen.

4.5.3.5 Grinding Wheels

Formed, resinoid-bonded, diamond-impregnated (minimum 320 grit in a resinoid bond) wheels may be necessary both to fabricate critical shapes (e.g., gage section transition radius) and to minimize grinding vibrations and subsurface damage in the test material. Formed wheels may require periodic dressing and shaping (truing), processes which can be done dynamically within the fabrication machine, to maintain the cutting and dimensional integrity.

4.6 Post-Machining Treatments

4.6.1 Heat Treatment

For specimens subjected to a multiaxial stress state, grinding causes the direction transverse to grinding to exhibit lower strength than the direction parallel to grinding. This effect can be alleviated by heat treating or etching to heal or blunt the machining damage, thereby promoting isotropic strength behavior.

5. ADDITIONAL GUIDANCE

5.1 Reference Documents

MIL-F-48616	Filter (Coatings), Infrared Interference, General Specification for
MIL-O-13830	Optical Components for Fire Control Instruments; General Specification Governing the Manufacture, Assembly, and Inspection of
ASTM C 1161	Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
ASTM C 1273	Test Method for Tensile Strength of Monolithic Advanced Ceramics at Ambient Temperature

ASTM C 1424 Test Method for Monotonic Compressive Strength of Advanced Ceramics at Ambient Temperature

ASTM C 1499 Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature

IEEE SI 10 Use of the International System of Units (SI): The Modern Metric System

Harris, D. C. (1999). *Materials for Infrared Windows and Domes.* Bellingham, WA: SPIE Optical Engineering Press

(Copies of the ASTM documents are available from ASTM International, 100 Bar Harbor Drive, W. Conshohocken, PA)

5.2 Intended Use

This recommended practice is intended for use in fabricating ceramic test specimens or engineering components when customary, application, or other machining procedures are not applicable or available.

5.3 Key Word Listing

advanced ceramic
diamond-grit
grinding
lapping
machining
polishing

APPENDIX A

RECOMMENDED POLISHING SPECIFICATIONS FOR CERAMIC WINDOWS

A.1 SCOPE

The specifications in this appendix may be used for polishing ceramic windows, but are not a mandatory part of this handbook. They are intended for additional information.

A.2 DEFINITIONS

The definitions of applicable terms used in this practice follow:

Bubble: An imperfection; a relatively large blister or gaseous inclusion.

Dig: A pit, bubble, inclusion that intersects a surface and is manifested as a deep, short scratch with a length-to-width ratio less than 5:1.

Inclusion: Any foreign matter or particles that are either encapsulated or imbedded in the main body.

Pit: Small crater in the surface with its width approximately the same order of magnitude as its depth.

Scratch: A shallow groove or cut below the established plane of the surface, with a length to width ratio greater than 5:1.

A.3 GENERAL GUIDANCE

Windows are often composed of fused silica, quartz, or sapphire and can be modeled as a mixed clamped/free support plate loaded by a pressure on one face. The resulting biaxial stress state is linearly distributed through the thickness cross section, reaching a maximum at the surface. Maximum surface stress leads to susceptibility of the fracture from surface flaws.

Surface flaws may be either intrinsic (e.g., pore, agglomerates intersecting the surface) or induced (e.g., foreign object damage or machining damage). The aim of this appendix is to ensure that induced surface flaws due to machining are minimized.

A.4 ADDITIONAL GUIDANCE

A.4.1 Example 1: Polishing Procedures for Sapphire Windows

The face of the window is to be the C-plane +/- 2°.

A.4.1.1 Rough Removal

Remove a minimum of 0.25 mm of material using double-sided lapping and a free-abrasive comprised of 20 to 40 µm boron carbide.

A.4.1.2 Grind

Circumferentially grind the edges and bevels with a 320 to 400 grit fixed diamond abrasive wheel. Free-abrasive lap the edges and bevels with grit sizes as follows. Make a best effort to remove approximately 3 times the grit size used in the previous stage. Break edges 0.02 to 0.1 mm simultaneously.

a. Remove 30 µm off each surface with 3 to 5µm grit size.

b. Remove 5 µm off each surface with 1 µm grit size.

A.4.1.3 Lap

Lap both faces with the grit sizes as follows. Remove approximately 3 times the grit size used in the previous stage by using diamond or boron nitride in a free abrasive mode. Ensure that a short finish is avoided and that the face contacting the lap plate is not scratched or indented by grit remaining on the lap plate.

a. If necessary, remove 100 µm off each surface with 9 µm grit size.

b. Remove 30 µm off each surface with 3 to 5 µm grit size.

c. Remove 10 µm off each surface with 1 µm grit size.

A.4.1.4 Anneal

Anneal at 1450 °C in air for >1 hour. Ensure that large test specimens or components do not sag. One way to prevent these effects is by placing them on a sapphire support surface.

A.4.1.5 Buff

Buff both faces by removing 0.0050 to 0.0125 mm with colloidal silica. Work benches, etc. can contain grit from operations, and polished pieces should not be placed on such surfaces. During handling, all polished pieces should be placed on a soft, clean surface such as cloth or foam padding.

A.4.2 Example 2: Low Damage Sapphire Windows

The following are fabrication procedures for low damage sapphire windows from as-cored rodstock.

A.4.2.1 Slice

Slice the rod-stock to produce blanks 1.25 mm over the finished size by using a 180-grit metal bond diamond cut-off blade. Clean the blanks and wax-mount them to a steel plate.

A.4.2.2 Blanchard Grinding

Remove 0.50 mm off each side by Blanchard grinding with a 220-grit metal bond wheel. Remove the rodstock from steel plate(s) and clean with OptiClear™ solvent, alcohol, and then acetone.

A.4.2.3 Loose-Abrasive Grind

Remove 0.125 mm by using double-sided loose-abrasive grind with 320-grit boron carbide. Clean the parts with water and alcohol. Wax-stack the parts.

A.4.2.4 Edge-Grind

Edge-grind the outer diameter (OD) by using a 220-grit diamond polyamide bond wheel. Hand polish OD edges to a semi-polished appearance by using copper sheathing with a 6- to 9- micron diamond slurry.

A.4.2.5 Grind

Grind the bevels (2 places) with a 220-grit polyamide bond wheel.

A.4.2.6 Brush Polish

Wax-mount the blanks to the steel plate. Brush polish the edges by using a 7-station planetary machine with 6- to 9- micron diamond. Reverse the parts and repeat the brush-polishing. operation for the second side of the blanks. Remove the blanks from the steel plates and clean the blanks with OptiClear™ solvent, alcohol, and then acetone.

A.4.2.7 Diamond Polishing

Wax-mount the blanks to the steel plate. Polish using a 2-step diamond polishing procedure. Polish using colloidal silica. Reverse the parts and repeat the polishing operations for the second side of the blanks. Keep track of the last side polished – this should be the tension side. Remove the blanks from the steel plates and clean the blanks with OptiClear™ solvent, alcohol, and acetone.

A.4.2.8 Etch

Etch the edges as necessary.

A.4.3 Example 3: Polishing Procedures for Fused Silica and Quartz Windows

A.4.3.1 Grind

Circumferentially grind the edges and bevels with a 320-grit or finer fixed diamond abrasive wheel.

A.4.3.2 Lap

Lap both faces with the grit sizes as follows (depending on the coarse grind):

a. Remove 200 µm off each surface with 30 µm grit size abrasive.

b. Remove 91 µm off each surface with 12 µm grit size abrasive.

c. Remove 38 µm off each surface with 5 µm grit size abrasive.

A 4.3.3 Wipe

Wipe all edges with diluted acid (40 percent HCL) for 50 min.

A.4.4 Example 4: Surface Finish Requirements

A.4.4.1 Scratch/Dig Specifications

An accepted but qualitative measure of surface finish for optical components is the scratch/dig specification detailed in MIL-O-13830 and MIL-F-48616. Table 1 summarizes the scratch/dig specifications for the two MIL specifications.

A.4.4.2 Parallelism and Flatness

Besides the usual tolerances and scratch dig specifications, additional specifications such as parallelism (e.g., 5 arc min) and flatness (2 waves at 0.6328 µm) should be included for optical applications.

A.4.4.3 Maximum Allowable Scratch/Dig Number

For sapphire windows, a maximum allowable scratch/dig number as defined by MIL-O-13830 is 60. For fused silica and quartz windows, a maximum allowable scratch/dig number as defined by MIL-O-13830 is 80.

Table 1—Scratch/Dig Specification Details

[1]MIL-O-13830			[2]MIL-F-48616				
Number	Maximum Scratch Width	Maximum Dig Diameter	Letter	Scratch Width	Dig Diameter	Disregard Scratch Width	Disregard Dig Diameter
10	1 µm	100 µm	A	5 µm	50 µm	<1 µm	≤10 µm
20	2 µm	200 µm	B	10 µm	100 µm	<2.5 µm	≤25 µm
40	4 µm	400 µm	C	20 µm	200 µm	<5 µm	≤50 µm
60	6 µm	600 µm	D	40 µm	300 µm	<10 µm	≤50 µm
80	8 µm	800 µm	E	60 µm	400 µm	<10 µm	≤100 µm
			F	80 µm	500 µm	<20 µm	≤100 µm
			G	120 µm	700 µm	<20 µm	≤200 µm
			H		1000 µm		≤250 µm

Notes:
[1]MIL-O-13830, Optical Components for Fire Control Instruments; General Specification Governing the Manufacture, Assembly, and Inspection of
[2]MIL-F-48616, Filter (Coatings), Infrared Interference, General Specification for