METRIC/SI

NASA TECHNICAL HANDBOOK

NASA-HDBK-6007B

Office of the NASA Chief Engineer

Approved: 2018-07-17
Superseding NASA-HDBK-6007A

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

DOCUMENT HISTORY LOG

Status	Document Revision	Change Number	Approval Date	Description
Baseline			2007-11-19	Initial Release
Revision	A		2013-12-02	General Revision
Revision	B		2018-07-17	Significant changes were made to this NASA Technical Handbook. It is recommended that it be reviewed in its entirety before implementation. Key changes were as follows: 1.1: A paragraph was added as an introduction to Appendix C. Appendix C, Examples of Handling and Machining Related Damage, was added.

FOREWORD

This NASA Technical Handbook is published by the National Aeronautics and Space Administration (NASA) as a guidance document to provide 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 NASA Technical Handbook is approved for use by NASA Headquarters and NASA Centers and Facilities. It may also apply to the Jet Propulsion Laboratory (a Federally Funded Research and Development Center (FFRDC)), other contractors, recipients of grants and cooperative agreements, and parties to other agreements only to the extent specified or referenced in applicable contracts, grants, or agreements.

NASA missions use brittle materials such as ceramics and glasses in applications such as optical components. Such components are required to sustain intermittent or continuous structural loads. The strength of components in such applications is dependent on the flaws, both inherent and induced. The size and corresponding strength of induced flaws depend on the material removal process and handling of test specimens or components.

This NASA Technical Handbook establishes guidelines and recommendations for machining of advanced ceramics and glasses. Machining of advanced ceramics and glasses is often necessary to achieve certain design requirements such as dimensional (tolerance) requirements, geometric shape, functional fit, and surface finish. However, operations such as surface grinding can cause a significant decrease in the strength of advanced ceramics and glasses 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 and glasses can apply as a starting point in specification writing and fabrication of ceramic and glass test specimens and components.

Requests for information should be submitted via “Feedback” at https://standards.nasa.gov. Requests for changes to this NASA Technical Handbook should be submitted via MSFC Form 4657, Change Request for a NASA Engineering Standard.

______Original signed by______
Ralph R. Roe, Jr.
NASA Chief Engineer

_________2018-07-17_________
Approval Date

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

1. SCOPE

1.1 Purpose

The purpose of this NASA Technical Handbook is to cover recommended material removal processes (i.e., machining or grinding) for advanced ceramics and glasses, which are referred to as “ceramics” in this NASA Technical Handbook. It is applicable to both test specimens and components, hereafter referred to as “specimens.” This NASA Technical Handbook is not intended to replace or supersede 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 NASA Technical Handbook 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, Recommended Polishing Specifications for Ceramic Windows, provides a specific application example.

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 NASA Technical 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 with the goal of minimizing subsurface damage.

Although careful visual inspection of components is recommended, it may or may not reveal inappropriate machining, handling and contaminant subsurface damage.

Handling of test specimens and components can introduce damage significantly in excess of the damage produced by common fabrication practices. Specimens and components must be handled carefully. Appendix C, Examples of Handling and Machining Related Damage, provides examples of damage and recommendations to avoid such problems.

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

1.2 Applicability

This NASA Technical Handbook is applicable to material removal processes (machining or grinding) for advanced ceramics and glasses, which are referred to as “ceramics” in this NASA Technical Handbook. It is applicable to both test specimens and components, which are referred to in this NASA Technical Handbook as “specimens.”

This NASA Technical Handbook is approved for use by NASA Headquarters and NASA Centers and Facilities. It may also apply to the Jet Propulsion Laboratory (a Federally Funded Research and Development Center (FFRDC)), other contractors, recipients of grants and cooperative agreements, and parties to other agreements only to the extent specified or referenced in their applicable contracts, grants, or agreements.

This NASA Technical Handbook, or portions thereof, may be referenced in contract, program, and other Agency documents for guidance.

The practice of material removal (machining or grinding) for advanced ceramics 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. Contact the Center's Occupational Health Office for guidance.

This NASA Technical Handbook is intended primarily for use with advanced ceramics and glasses 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; so application of this practice may not be appropriate.

Values expressed in this NASA Technical Handbook are in accordance with IEEE/ASTM SI 10™-2016, American National Standard for Metric Practice.

2. APPLICABLE DOCUMENTS

2.1 General

The documents listed in this section are applicable to the guidance in this NASA Technical Handbook.

2.1.1 The latest issuances of cited documents apply unless specific versions are designated.

2.1.2 Non-use of a specifically designated version is approved by the delegated Technical Authority.

Applicable documents may be accessed at https://standards.nasa.gov or obtained directly from the Standards Developing Body or other document distributors. When not available from these sources, information for obtaining the document is provided.

2.2 Government Documents

None.

2.3 Non-Government Documents

J. A. Salem, N. N. Nemeth, L. M. Powers, and S. R. Choi (1996). *Reliability Analysis of Uniaxially Ground Brittle Materials*, Journal of Engineering for Gas Turbines and Power, Trans. of the ASME, Vol. 118, pp. 863-871

ASTM International

ASTM C1161	Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
ASTM C1239	Standard Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics
ASTM C1273	Standard Test Method for Tensile Strength of Monolithic Advanced Ceramics at Ambient Temperatures
ASTM C1424	Standard Test Method for Monotonic Compressive Strength of Advanced Ceramics at Ambient Temperature
ASTM C1495	Standard Test Method for Effect of Surface Grinding on Flexure Strength of Advanced Ceramics
ASTM C1499	Standard Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature

Co-Sponsored by Institute of Electrical and Electronics Engineers (IEEE) and ASTM International

IEEE/ASTM SI 10™ 2016	American National Standard for Metric Practice

Reference documents are provided in Appendix B.

2.4 Order of Precedence

2.4.1 The guidance established in this NASA Technical Handbook does not supersede or waive existing guidance found in other Agency documentation.

2.4.2 Conflicts between this NASA Technical Handbook and other documents are resolved by the delegated Technical Authority.

3. ACRONYMS, ABBREVIATIONS, SYMBOLS, AND DEFINITIONS

3.1 Acronyms, Abbreviations, and Symbols

°	degrees
>	greater than
<	less than
≤	less than or equal to
±	plus or minus
~	weak approximation
µm	micron
ASTM	American Society for Testing and Materials
C	Celsius
CFCCs	continuous fiber ceramic composites
FFRDC	Federally Funded Research and Development Center
HDBK	Handbook
IEEE	Institute of Electrical and Electronics Engineers
kPa	kilopascal
m	meter
MIL	military
min	minute
mm	millimeter
NASA	National Aeronautics and Space Administration
OD	outer diameter
sec	second
SI	International System of Units
X	times sign

3.2 Definitions

The following definitions of applicable terms are taken from ASTM C1145, Standard Terminology of Advanced Ceramics, or are specific to this NASA Technical Handbook:

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

Extraneous Flaws: Strength-limiting flaws introduced on the surface of test specimens or the component being designed.

Note: 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.

Note: Fractography can be used to determine the nature and location of the critical fracture origin.

Intrinsic Flaws: Strength-limiting flaws that exist throughout the volume of a test specimen or component.

Note: Examples are pores and agglomerations that are formed during processing and consolidation of the advanced ceramic.

Machining Damage: As used in fractography, chips and surface or 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 ceramics literature, "slow crack growth curve" is often called a "static fatigue" curve.

4. GUIDANCE

4.1 Significance and Use

4.1.1 Extraneous Flaws

Generally, strength distributions of ceramics are probabilistic and can be described by a weakest link failure theory. (See ASTM C1239, Standard Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics.) 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.

4.1.2 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 that 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 NASA Technical Handbook 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 gauge 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.

For verification, fractographic examination of tested baseline specimens is used to ascertain the level of machining damage at the fracture origin. In some instances, undetected grinding-induced damage may combine or join with the inherent flaw that acts as a hybrid source or origin of fracture. (Refer to Salem, et. al., 1996.) This may impose a negative bias on the measured strength result. Fractographic analysis of broken specimens is highly recommended to characterize the types, locations, and sizes of fracture origins as well as to detect stable crack extension due to slow crack growth.

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 gauge 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. ASTM C1495, Standard Test Method for Effect of Surface Grinding on Flexure Strength of Advanced Ceramics, gives some guidance on grinding machines and wheels.

4.4 Precautionary Statement

4.4.1 Dust as a Health Hazard

Grinding and cutting advanced ceramics often create fine particles that may be a health hazard. Materials containing whiskers, small fibers, or silica particles may also cause health hazards when tested. For such materials, the operator is advised to consult the Center's Occupational Health office and safety data sheet for guidance prior to testing. Potential exposure monitoring along with suitable ventilation or respiratory protective equipment may be warranted.

4.5 Recommended Procedures for Specific Configurations

4.5.1 Prismatic Uniaxial Flexure Bars

This section is, for example, the standard procedure used in ASTM C1161, Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature. Application-matched procedures are also allowed.

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.

> Note: Although most ceramics should be machined wet, some ceramics such as boron nitrides should be machined dry using air as a coolant to avoid adverse reactions. If in doubt, consult the manufacturer's recommendations.

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, Horizontal-Spindle Surface Grinding for Machining Prismatic Uniaxial Flexure Bars. Do not use Blanchard or rotary grinding. (See Figure 2, Vertical-Spindle Surface (Blanchard) Grinding and Polishing for Machining Biaxial Flexure Disks.)

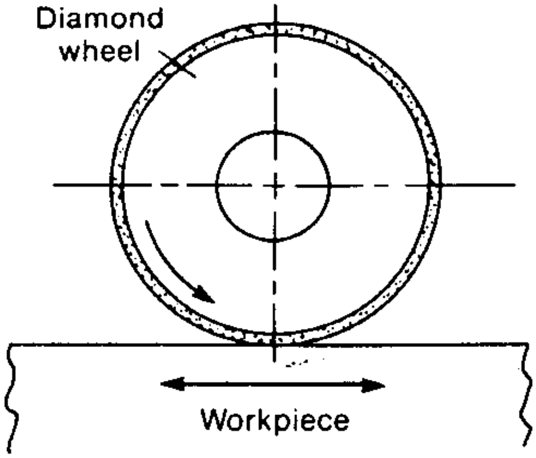

Figure 1—Horizontal-Spindle Surface Grinding for Machining Prismatic Uniaxial Flexure Bars

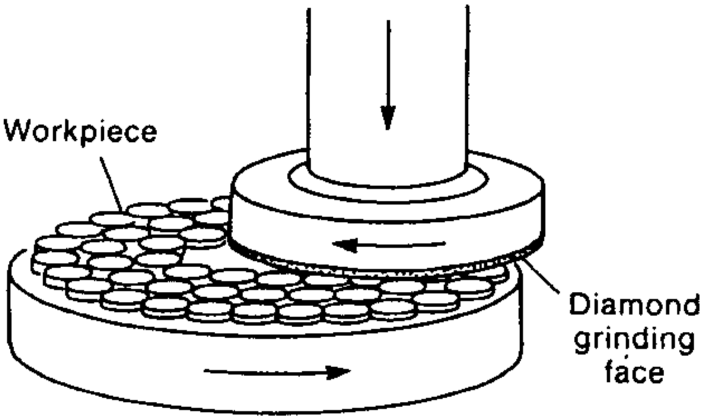

Figure 2—Vertical-Spindle Surface (Blanchard) Grinding and Polishing for Machining Biaxial Flexure Disks

4.5.1.2 Stock Removal Rate

The stock-removal rate should not exceed 0.03 millimeter (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. Wheel speed should not be less than 25 meter per second (m/sec). Table speeds should not be greater than 0.25 m/sec.

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 round them 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. A chamfer can also be added by hand sanding parallel to the long axis of the specimen. For uniaxial bend specimens, no chipping is allowed. Up to 50 X magnification may be used for verification.

4.5.2 Flat Biaxial Flexure Disks

This section is, for example, as that used in ASTM C1499, Standard Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature.

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.

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 mitigate 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 micron (µm) diamond pastes for ~30, ~25, and ~15 minute (min), respectively. For tungsten carbide, successive machine lapping with 15 and 6 µm diamond pastes for ~60 and ~30 min, respectively, with a pressure of ~13.8 kilopascals (kPa) was sufficient. Specific procedures need to be developed for other materials.

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 this section without otherwise altering the material's strength. However, note that annealing can significantly alter a material's properties; and specific procedures need to be developed for each material. Soda-lime silicate glass can be annealed by heating at 520°C for 2 hours followed by slow cooling.

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

This section is, for example, as that used in ASTM C1273, Standard Test Method for Tensile Strength of Monolithic Advanced Ceramics at Ambient Temperatures; and ASTM C1424, Standard Test Method for Monotonic Compressive Strength of Advanced Ceramics at Ambient Temperature.

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, Outside Diameter Cylindrical Grinding for Machining Cylindrical Tension/Compression Rods.)

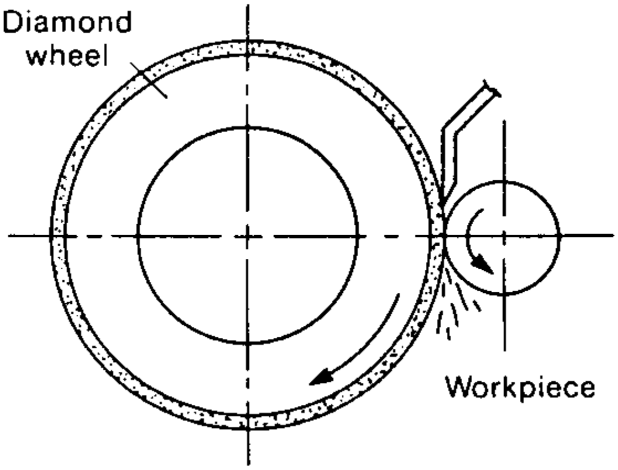

Figure 3—Outside Diameter Cylindrical Grinding for Machining Cylindrical Tension/Compression Rods

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.

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 gauge section. Such a final longitudinal grinding operation is not necessary for compressive strength test specimens because of the volume-related (that is, not surface-related) compressive strength mechanism.

4.5.3.4 Specimen Mounting

Generally, computer numerical control 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., gauge 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 that 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.

APPENDIX A

RECOMMENDED POLISHING SPECIFICATIONS FOR CERAMIC WINDOWS

A.1 Scope

The guidance in this Appendix may be used for polishing ceramic windows but is not a mandatory part of this NASA Technical Handbook. It is intended for additional information. For complex shapes such as lenses in aerospace optical systems, precision diamond turning is applicable but requires more sophisticated equipment than traditional grinding and polishing methods. (See NASA Lessons Learned Information System (LLIS) Database Entry 755 for more information.)

A.2 Definitions

The definitions of terms used in Appendix A of this NASA Technical Handbook are as follows:

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 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). Induced flaws are also referred to as extrinsic flaws. 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:

a. Remove 0.030 mm off each surface with 3 to 5 µm abrasive size.

b. Remove 0.005 mm off each surface with 1 µm abrasive size.

Make a best effort to remove approximately three times the abrasive size used in the previous stage. Break edges 0.02 to 0.1 mm, simultaneously.

A.4.1.3 Lap

Lap both faces with the grit sizes as follows:

a. If necessary, remove 0.100 mm off each surface with 9 µm size abrasive.

b. Remove 0.030 mm off each surface with 3 to 5 µm size abrasive .

c. Remove 0.010 mm off each surface with 1 µm size abrasive.

Remove approximately three times the abrasive 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.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 0.005 to 0.013 mm with colloidal silica. Work surfaces 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 sections provide 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 an optical cleaning suspension 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-µm 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-µm 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 an optical cleaning suspension 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, which should be the tension side. Remove the blanks from the steel plates and clean the blanks with an optical cleaning suspension 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 0.200 mm off each surface with 30 µm size abrasive.

b. Remove 0.091 mm off each surface with 12 µm size abrasive.

c. Remove 0.038 mm off each surface with 5 µm size abrasive.

A.4.3.3 Wipe

Wipe all edges with diluted acid (40 percent hydrofluoric acid) for 15 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 in general is the scratch/dig specification detailed in MIL-PRF-13830B, Optical Components for Fire Control Instruments; General Specification Governing the Manufacture, Assembly, and Inspection of, and MIL-F-48616, Filter (Coatings), Infrared Interference, General Specification For. Table 1, Scratch/Dig Specification Details, summarizes the scratch/dig specifications for the two military specifications.

Table 1—Scratch/Dig Specification Details

MIL-PRF-13830B			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

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-PRF-13830B is 60. For fused silica and quartz windows, a maximum allowable scratch/dig number as defined by MIL-PRF-13830B is 80. Scratch and dig specifications are often different (e.g., an 80/40 number).

APPENDIX B

REFERENCES

B.1 Purpose

This Appendix provides documents available for reference.

B.2 Reference Documents

ASTM C242	Standard Terminology of Ceramic Whitewares and Related Products
ASTM C1145	Standard Terminology of Advanced Ceramics
ASTM C1322	Standard Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics
ASTM F109	Standard Terminology Relating to Surface Imperfections on Ceramics
NASA/TM-2006-214023	Slow Crack Growth and Fracture Toughness of Sapphire for the International Space Station Fluids and Combustion Facility
NASA/TM-2009-215802	Failure Analysis of Sapphire Refractive Secondary Concentrators
MIL-F-48616	Filter (Coatings), Infrared Interference, General Specification for (Inactive for New Design)
MIL-PRF-13830B	Optical Components for Fire Control Instruments; General Specification Governing the Manufacture, Assembly, and Inspection of

APPENDIX C

EXAMPLES OF HANDLING AND MACHINING RELATED DAMAGE

C.1 Purpose

This Appendix provides examples of handling- and machining-related damage on the behavior of ceramic test specimens and structural components. (See lessons learned on handling of ceramic electronic components in LLIS Database Entry 18502.)

The adverse effects of machining, handling, and clamping on the behavior of ceramic and glass structural components are given in the following examples. Rules-of-thumb for working with such materials are as follows:

1. Machine and handle specimens and components appropriately and carefully.

2. Handle with care; do not strike, scratch, or rub with tooling of any kind because it will scratch and crack ceramics despite high hardness. Do not press or slide against hard surfaces.

3. Avoid mechanical clamping with hard contacts as it will induce cracking and chipping. Use compliant layers (e.g., seals) or braze.

4. Measure with care. Non-contacting methods are preferred in many cases, particularly for polished surfaces. Ball or point micrometers should not be used. Calipers and flat anvil micrometers also can cause damage, especially if over-tightened or wedged against the specimen during removal (see (b) in Figure 4, Damage to a Ceramic Crystal: (a) Tool Chip on Bevel; (b) Micrometer Edge Damage; (c) Tool Scratch). In some cases, measurements can be made after testing.

C.2 Strength Reduction due to Handling

The damage shown in Figure 4 (a through c) resulted in a lower characteristic strength (35 MPa) as compared to that of well-machined and handled specimens (58 MPa), substantially improving the margin of safety.

Figure 4—Damage to a Ceramic Crystal: (a) Tool Chip on Bevel; (b) Micrometer Edge Damage; (c) Tool Scratch

C.3 High Temperature, High Pressure Water Cell

Sapphire windows on a test cell showed rapid degradation (ten use cycles) and went from clear to scratched or pitted. (See Figure 5, Pitting and Scratching Apparent After High Temperature, High Pressure Testing of Sapphire Windows.) The window vendor thought the scratches might be due to mechanical damage in the test cell via particles, despite no particulate matter existing in the closed system. The problem was solved via better polishing and thermal treatment that eliminated subsurface polishing damage, thereby leading to substantially improved life. (See NASA/TM-2006-214023, Slow Crack Growth and Fracture Toughness of Sapphire for the International Space Station Fluids and Combustion Facility.)

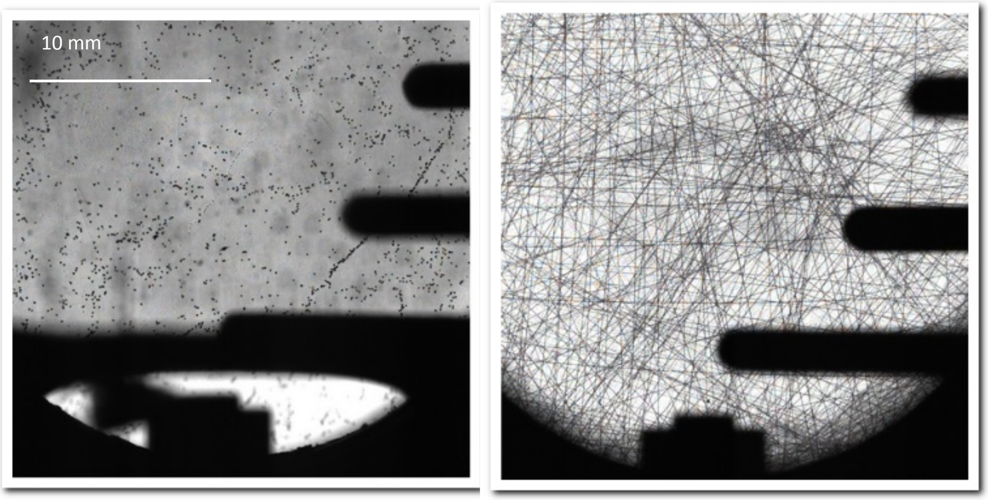

Figure 5—Pitting and Scratching Apparent After High Temperature, High Pressure Testing of Sapphire Windows

C.4 Solar Concentrator Failure

Testing of single crystal solar concentrators for providing thermal energy to space systems resulted in unexpected, catastrophic failures. (See Figure 6, Handling Damage to a Sapphire Solar Concentrator Lens (NASA/TM-2009-215802, Failure Analysis of Sapphire Refractive Secondary Concentrators)). The failures were traced to surface damage on the concentrator lenses. In one case, the lens was scratched via a hard tool; in the other case, the lens face was impacted by a hard object. Better handling could have avoided the failures.

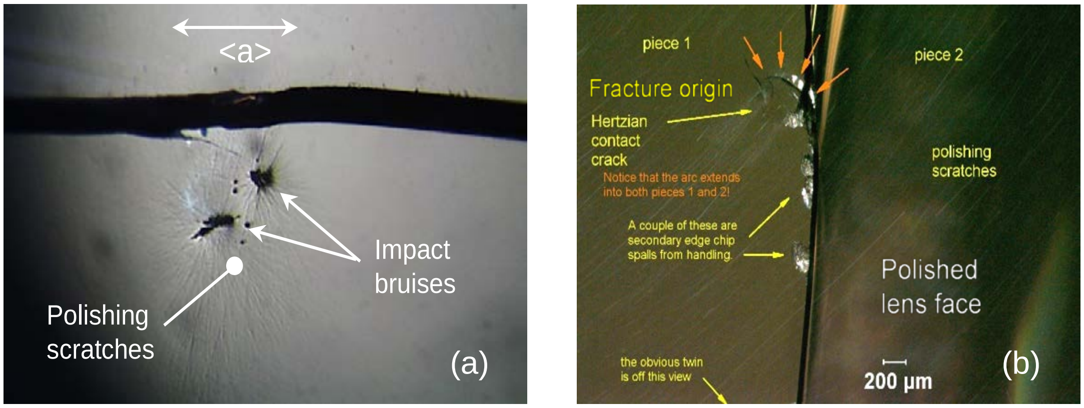

Figure 6—Handling Damage to a Sapphire Solar Concentrator Lens (Figures 10 ans 12 of NASA/TM-2009-215802)

C.5 Cracked Lens

A transparent ceramic window unintentionally came in contact with the hard edge of a misaligned clamp and was cracked in multiple locations. Better clamp design that allowed conformance between clamp and lens solved the problem. (See Figure 7, (a) Cracks Extending Through Lens; (b) Crack Partially Through Lens.)

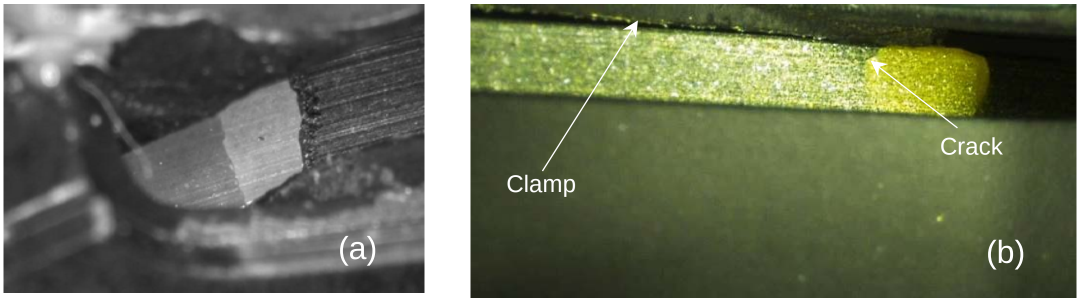

Figure 7—(a) Cracks Extending Through Lens; (b) Crack Partially Through Lens