NISTIR 4864

High-Temperature Flexure Fixture for Advanced Ceramics

George D. Quinn

U.S. DEPARTMENT OF COMMERCE
Technology Administration
National Institute of Standards and Technology
Ceramics Division
Gaithersburg, MD 20899

NIST

NISTIR 4864

High-Temperature Flexure Fixture for Advanced Ceramics

George D. Quinn

U.S. DEPARTMENT OF COMMERCE
Technology Administration
National Institute of Standards and Technology
Ceramics Division
Gaithersburg, MD 20899

June 1992

U.S. DEPARTMENT OF COMMERCE
Barbara Hackman Franklin, Secretary

TECHNOLOGY ADMINISTRATION
Robert M. White, Under Secretary for Technology

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
John W. Lyons, Director

HIGH-TEMPERATURE FLEXURE FIXTURE FOR ADVANCED CERAMICS

George Quinn
Ceramics Division

ABSTRACT

A test fixture for elevated-temperature flexure strength testing is presented. The fixture is suitable for fast-fracture or stress rupture experiments up to 1500 ^{o}C in air or inert environment.

INTRODUCTION

This report presents a four-point flexure fixture that meets the requirements of the standards MIL STD 1942 and ASTM C-1211 [1,2]. The fixture has spans of 20 and 40 mm and is designed to be used with "B" sized, 3 x 4 x 45-50 mm specimens, although other sizes are feasible.

An important requirement of the standards is that the load application rollers must be free to roll to relieve friction constraint. Standards that have been prepared in Europe also have this requirement [3,4]. Friction errors have been demonstrated in elevated-temperature flexure testing, both in fast-fracture and stress rupture modes of testing [5,6].

The fixture is a semiarticulating design for flat and parallel specimens. It is not necessary to have full articulation if specimens are well-machined or as-fired within reasonable parallelism limits. The fixture itself must also have parallel loading pins. Warped or twisted specimens (often in the as-fired state) may require the use of fully-articulating fixtures, but these also must have rolling-loading pins.

Either of two possible Base Plates can be used depending upon the style of test furnace. A large Base Plate (as shown in Figures 1-3) can be used for box type firebrick furnaces in order to distribute load over a large area. An alternative Base Plate (Figure 4) is more suitable for cylindrical furnaces equipped with pushrods or pedestals.

The fixture uses simple block shapes which are easy to machine and to assemble and join together. The pieces are interchangeable and can be replaced in the event of contamination or breakage. The fixture is thus more cost effective than schemes which have complicated slots cut into a single block. Such latter fixtures are prone to breakage and are difficult or impossible to repair if damaged.

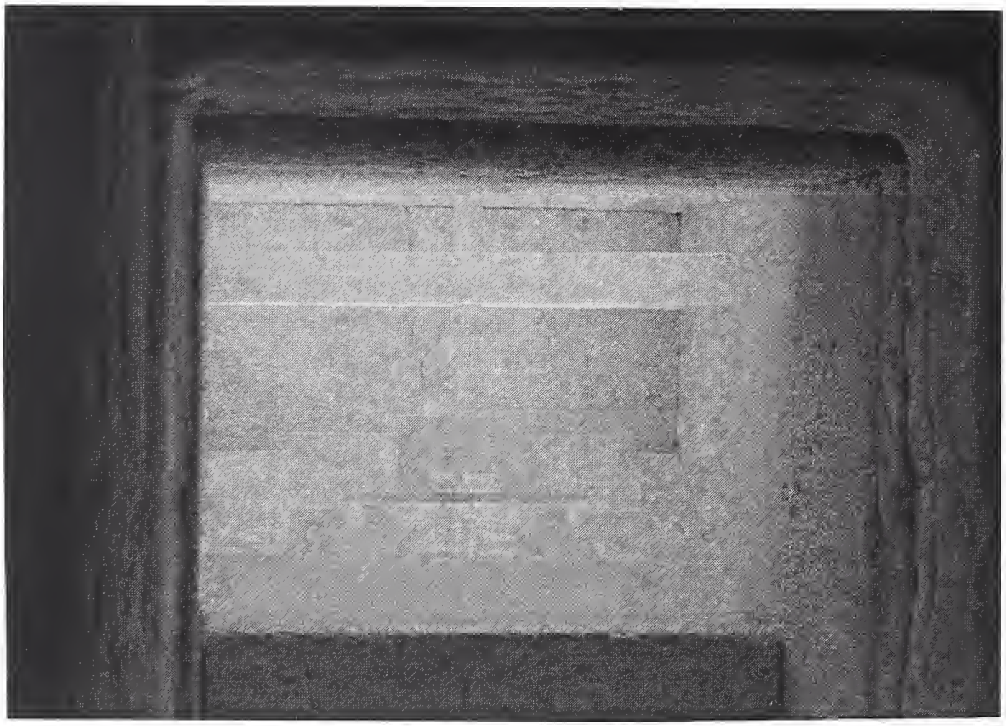


Figure 1. High-temperature flexure fixture in a resistance heating element furnace. Specimens can be either hot- or cold-loaded.

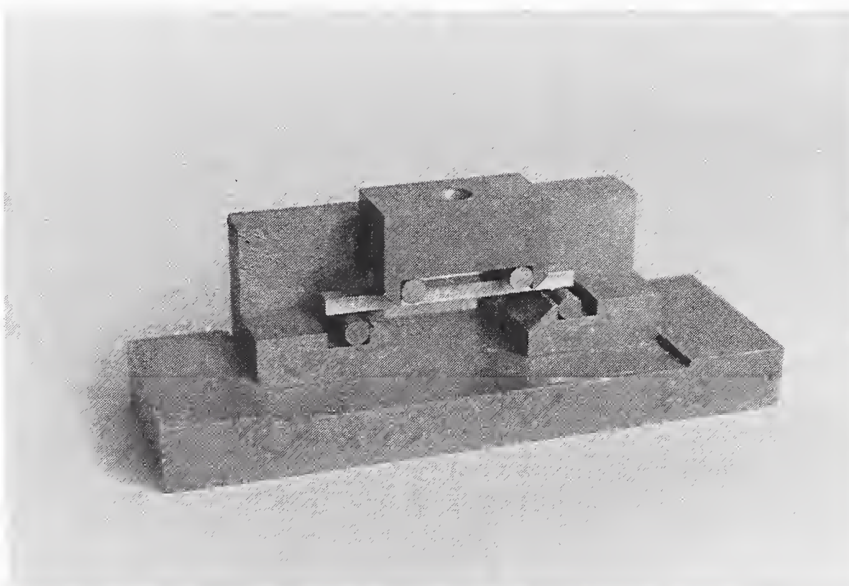


Figure 2. High-temperature flexure fixture with the large Base Plate. A smaller Base Plate is shown in Figure 4.

FLEXURE FIXTURE

Figures 1 and 2 show the fixture with the optional large Base Plate. The fixture is made of a combination of hot-pressed and sintered silicon carbide parts shown in Figures 3-9. The sintered silicon carbide pieces are more chemically inert, and less likely to react with a specimen or to bond together. The fracture toughness of sintered silicon carbide is too low to be used for all parts, however, (lest they shatter too easily) and there are some pieces that should be bonded together. The hot-pressed silicon carbide parts can be easily bonded and are more shatter resistant.

Some pieces are machined out of a solid billet of hot-pressed silicon carbide. This material has good strength, oxidation resistance, thermal shock and creep resistance and, in general, with ordinary care is resistant to breakage. The hot-pressed form is fabricated with a sintering aid (often 1-2% alumina) which will form a thin, coherent oxide layer that is useful for bonding the pieces together. Commercial grades of such material are Norton grade NC-203[*] and Ceradyne grade 146[**] hot-pressed silicon carbide.

Other parts, such as the Rollers, the Upper Loading Block and the Lower Roller Supports should be made of a purer form of silicon carbide that is less apt to bond together. Several forms of sintered silicon carbide, such as Carborundum's grade SA[***] or ESK's sintered SiC[****] are suitable. If breakage of the Upper Loading Block is a problem, then hot-pressed silicon carbide can be used.

The Base Plate, the Guide Blocks, and the Lower Roller Supports are carefully assembled and held together temporarily by small drops of an acetate household cement (e.g., Duco[#] or Elmers[##] "Clear Household" cement). Since the shapes are simple it is quite easy to do this such that fixture alignment and accuracy is well maintained. The cement drops should be applied in a tack-bonding fashion (along the edges) and should be never applied between two mating surfaces. Apply the tack drops in a systematic fashion, lest uneven shrinkage occurs which could cause parts to shift.

The fixture should then be fired in air at 1500 °C for 4-12 hours. The oxidation that will occur in the hot-pressed pieces will bond the pieces together. (Sintered pieces will not bond to each other.) The household acetate glue burns off leaving negligible residue. Its role is to keep the pieces aligned during handling and insertion into the furnace.

* Norton Co., Worcester, MA. The identification of this and the following materials does not indicate endorsement by the National Institute of Standards and Technology.

** Ceradyne Inc., Costa Mesa, CA.

*** Carborundum Co., Niagara Falls, NY.

**** ESK, Kempten, Germany, or New Caanan, CT.

\# Loctite, Cleveland, OH.

\## Borden, Inc. Columbus, OH.

Of course, the pushrods, the Upper Loading Block and the Loading Rollers should not be bonded. Specimen positioning shims (not shown) can be bonded if preferred.

The bonding process can be repeated if a piece subsequently comes loose. Excessive bondings may cause the sintering aid to deplete, however, requiring longer firing times or higher bonding temperatures (25 °C increments only!). If a piece bonds out of position or must be replaced due to breakage or contamination, it can be dislodged usually by a gentle hammer tap onto an intervening block of wood. Never directly impact the ceramic. Use silicon carbide abrasive paper (200-280 grit) to clean the surfaces and repeat the firing. If after repeated bondings, the sintering aid is depleted from the fixtures, try an alumina paste or cement along the seams. If this doesn't work, try alumina mixed with a small amount (< 20% by weight) of silica which will form some mullite. Apply this sparingly at the seams or along the mating surfaces.

The fixtures should be frequently cleaned. Never clean or scrape with any metallic tool. Slight burrs will react with the ceramic and form contaminating glasses (often rust-red colored). The fixture should be cleaned only with 200 or 280 grit silicon carbide (black) paper. A spare silicon carbide block or bend bar (which can be used to scape the grooves and slots). Avoid using alumina abrasive paper. Clean the Loading Rollers, the Lower Roller Supports and the Upper Loading Block, and pushrods. Do not clean the thin, glossy and coherent oxide layer on the hot-pressed parts since it is beneficial. Do not be alarmed by an iridescent sheen on the sintered parts since it is a normal, very thin oxide layer. Regard any blisters or discolored spots with suspicion, as possible furnace contamination. As a last resort, a major cleaning can be accomplished by sand-blasting the fixture.

The fixture is made of strong, dense, refractory grades of silicon carbide, but can be used with pushrods made of any grade silicon carbide or even alumina. Long pushrods can be fabricated from sintered silicon carbide, but may be prone to breakage. Extruded and sintered 99% alumina pushrods are cheap but will tend to creep-buckle. Recrystallized silicon carbide rods are also cheap, but their lower density will cause localized crushing that must be monitored. Siliconized silicon carbide pushrods are inexpensive and strong, but should not be used above the melting temperature of silicon (~1420 °C).

Holes for pushrod extensometry for creep measurements can be diamond drilled in the baseplate, but will cost about $200-300 (at 1992 rates) per hole.

COST

A single fixture set, with a spare Lower Roller Support and Loading Rollers will cost between $1500-2500 for machining.

Some machining venders may have the silicon carbide materials on hand. Otherwise, silicon carbide may have to be obtained from a ceramic company. The hot-pressed silicon carbide is suitable in the form of a flat plate, about 15 x 15 x (1.6-1.8) cm. A plate of this thickness is optimum since it minimizes the grinding and slicing costs. Such a plate will cost $700-1500. A smaller sintered silicon carbide plate is sufficient since only a few pieces are made from this material, but it also should be no less than 1.6 cm thick.

TEST SET UP

Some means must be provided to position the fixture in the center of the furnace, with particular attention to centering the fixture under the upper pushrod. Three ways of imparting load to the fixture are shown in Figure 10. If a rounded nipple exists on the top of the Upper Loading Block, then any flat loading ram should suffice to apply a well-centered load, and precise positioning of the fixture assembly is less important. The alternative Upper Loading Block with the spherical recess is much more sensitive to fixture positioning if it is used with a pushrod having a matching rounded tip. A simple alternative is to use a ceramic ball as shown in Figure 10c.

The fixture relies upon the two Guide Blocks to align the Upper Loading Block. This ensures that the 20 mm upper span is well centered over the 40 mm support span. The attached drawings show the two Guide Blocks as having the same groove dimension. It is alternatively possible to machine one with a wider groove. This eliminates any chance of binding. (Of course, during alignment, the Upper Loading Block would have to be positioned against the correct shoulder.) Small shim pieces (not shown) can be placed upon the Lower Roller Supports in order to position the specimen at the correct location (front-to-back) on the Rollers.

The important and tricky step is to get the Loading Rollers in their correct positions. There are several ways of doing this depending upon the furnace type.

1. *Cold-Furnace Installation.* The fixture parts can be tack bonded with the acetate household cement and inserted as a unit.

2. *Hot-Furnace Installation.* If the fixtures can be assembled outside the furnace and then inserted into the furnace, then try procedure 1. The glue will take a minute or more to burn off giving a chance to get the fixture in place.

If the furnace must be loaded hot, and the fixture must remain hot, then try Shims (drawings enclosed) to hold the lower Loading Rollers in their slots. Use long stainless steel tweezers (up to 30 cm are commercially available) with gloves and face shields. (If the furnace permits partial retraction of the fixture, then take advantage of this to warm load the fixture.) Remove the Upper

Load Block and affix the Upper Load Rollers in place with the acetate household cement. Deftly (!) insert the Upper Load Block and Rollers as an assembly into the furnace and position it on the specimen, and up against the Guide Blocks. The Guide Blocks will align the Load Block very quickly and easily. After preloading a little bit, remove the Shims from the Lower Roller Supports.

A light preload is recommended (<10% of the expected fracture load) but care should be taken not to let the load get too high during subsequent furnace heatup.

The hot-furnace loading sounds tricky, but with practice can be done without too much trouble depending upon the operator's dexterity, the style of furnace, and test temperature. A number of laboratories do it on a routine basis!

CONCLUSION

A simple fixture for measuring the flexural strength of advanced ceramics at elevated temperature is presented. It is a semiarticulating design to be used with flat and parallel specimens. The fixture meets the requirements of several standard test methods, and when used in accordance with these standards, will measure accurately flexure strengths to within 1%.

ACKNOWLEDGEMENT

Partial support for this project was provided by the Ceramic Technology Project, U. S. Department of Energy, Office of Transportation Technologies, under contract DE-AC-05-840R21400 with Martin Marietta Energy Systems, Inc.

REFERENCES

1. MIL STD 1942 (MR) and MIL STD 1942 (A), "Flexural Strength of High Performance Ceramics at Ambient Temperature," Available from Standardization Documents, Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094. (The A version is a 1990 update of the earlier 1983 MR version.
2. C-1211-90, "Standard Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperature," Annual Book of ASTM Standards, Vol. 15.01, ASTM, Philadelphia, PA, 1992.
3. NMP 51-110, Part 2, "Testing of Ceramic High Performance Materials, Four-point Flexure at Elevated Temperature," German Standards Institute, Berlin, 1991.
4. ENV, "Test Methods for Advanced Monolithic Technical Ceramics, Thermomechanical Properties, Part 1: Flexural Strength Tests," European Standard, 1992.
5. G. Quinn, "Twisting and Friction Errors in Flexure Testing," Ceram. Eng. and Sci. Proc., July-Aug., 1992, pp. 319-330.
6. G. Quinn, "Refinements to Flexure Testing," to be published in J. Eur. Ceram. Soc., 1992.

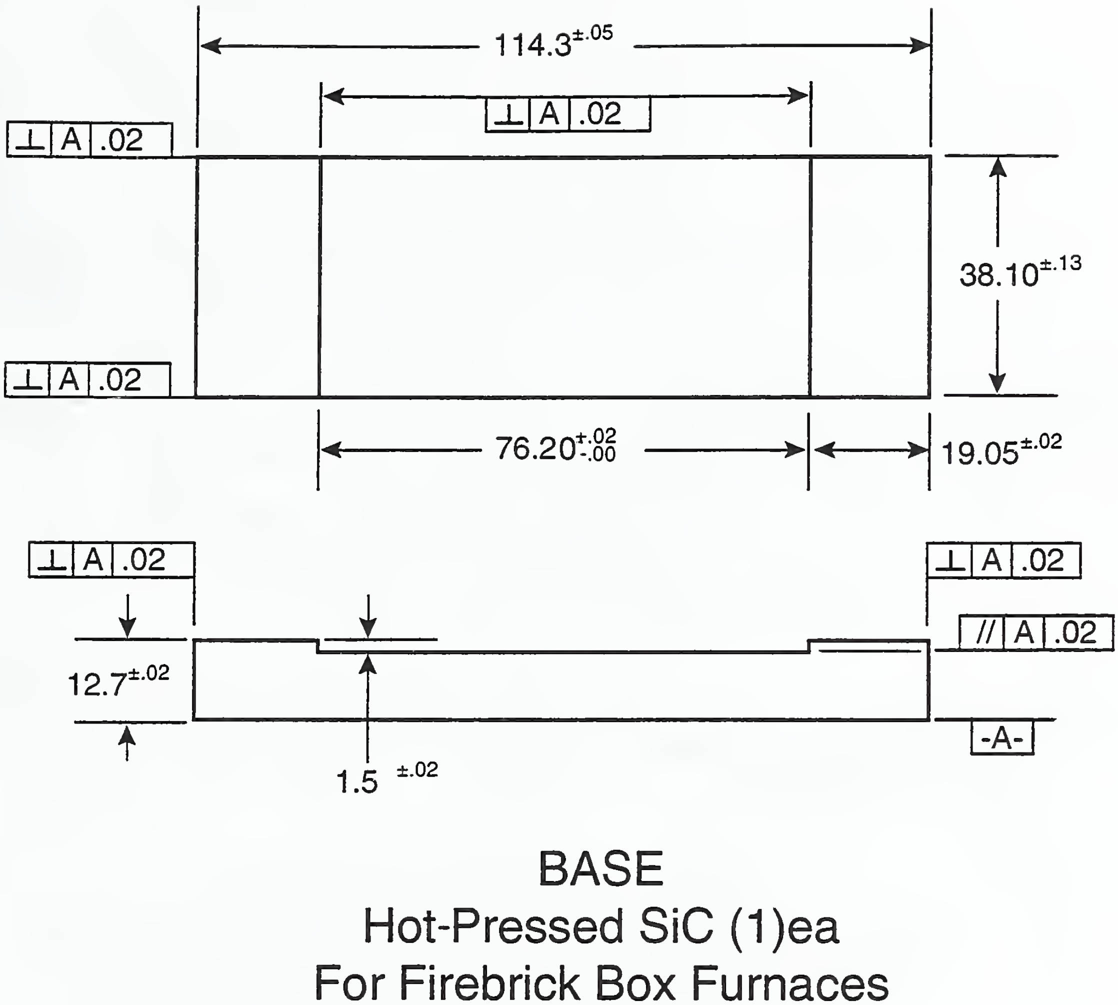
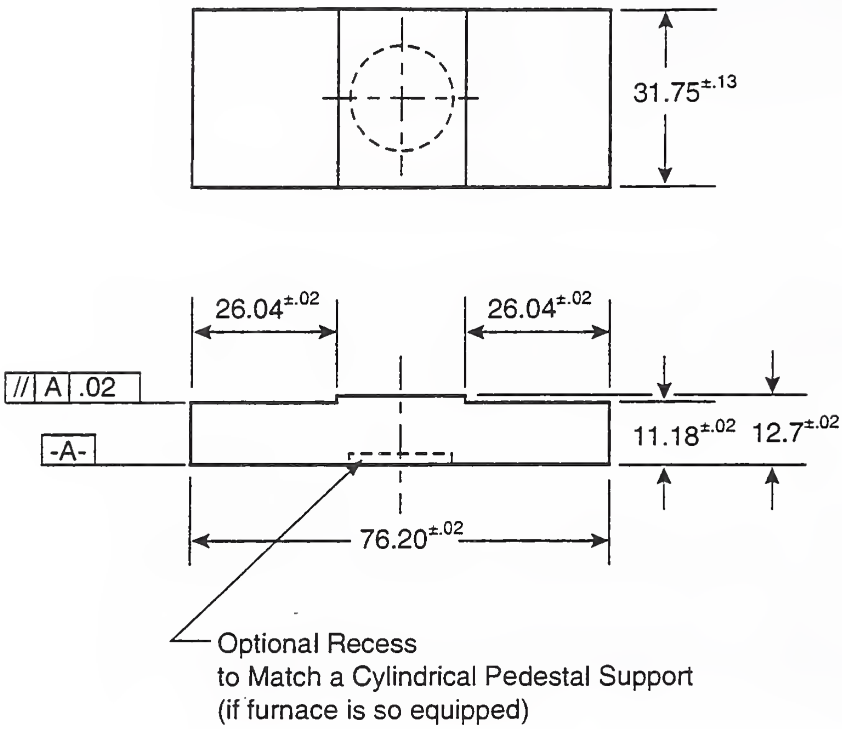
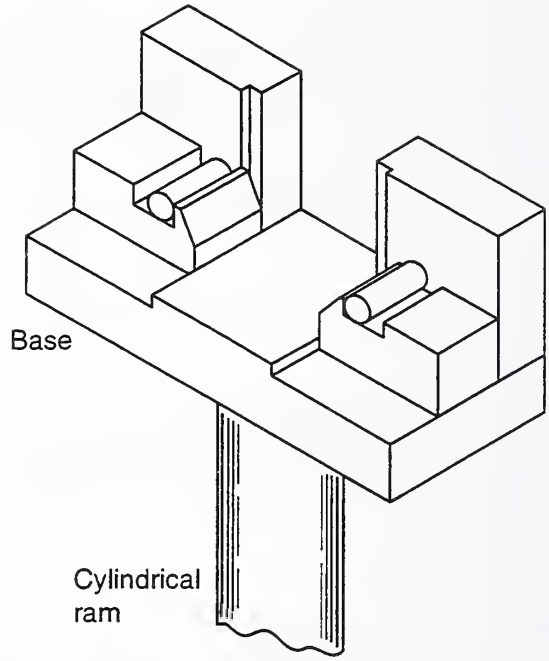


Figure 3. Base Plate of fixture suitable for firebrick furnaces. All dimensions are in mm.





Hot-Pressed
Silicon Carbide
(1) ea

BASE - ALTERNATIVE STYLE
FOR CYLINDRICAL FURNACES OR FURNACES
WITH CYLINDRICAL RAMS OR PEDESTALS

Figure 4. An alternative Base Plate suitable for pedestal or ram equipped furnaces. All dimensions in mm.

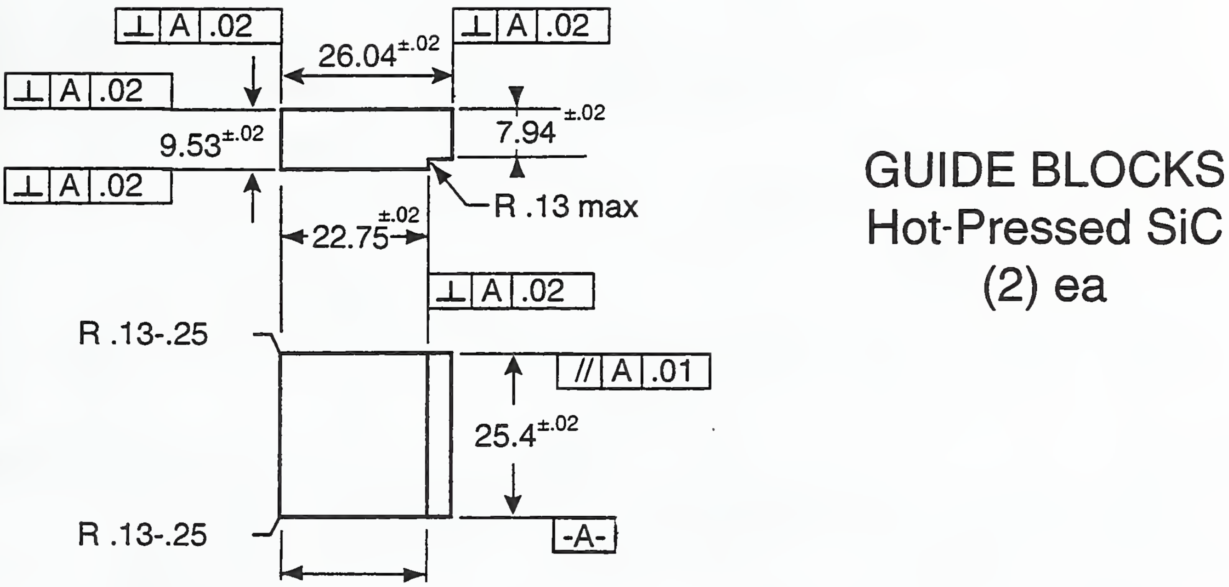
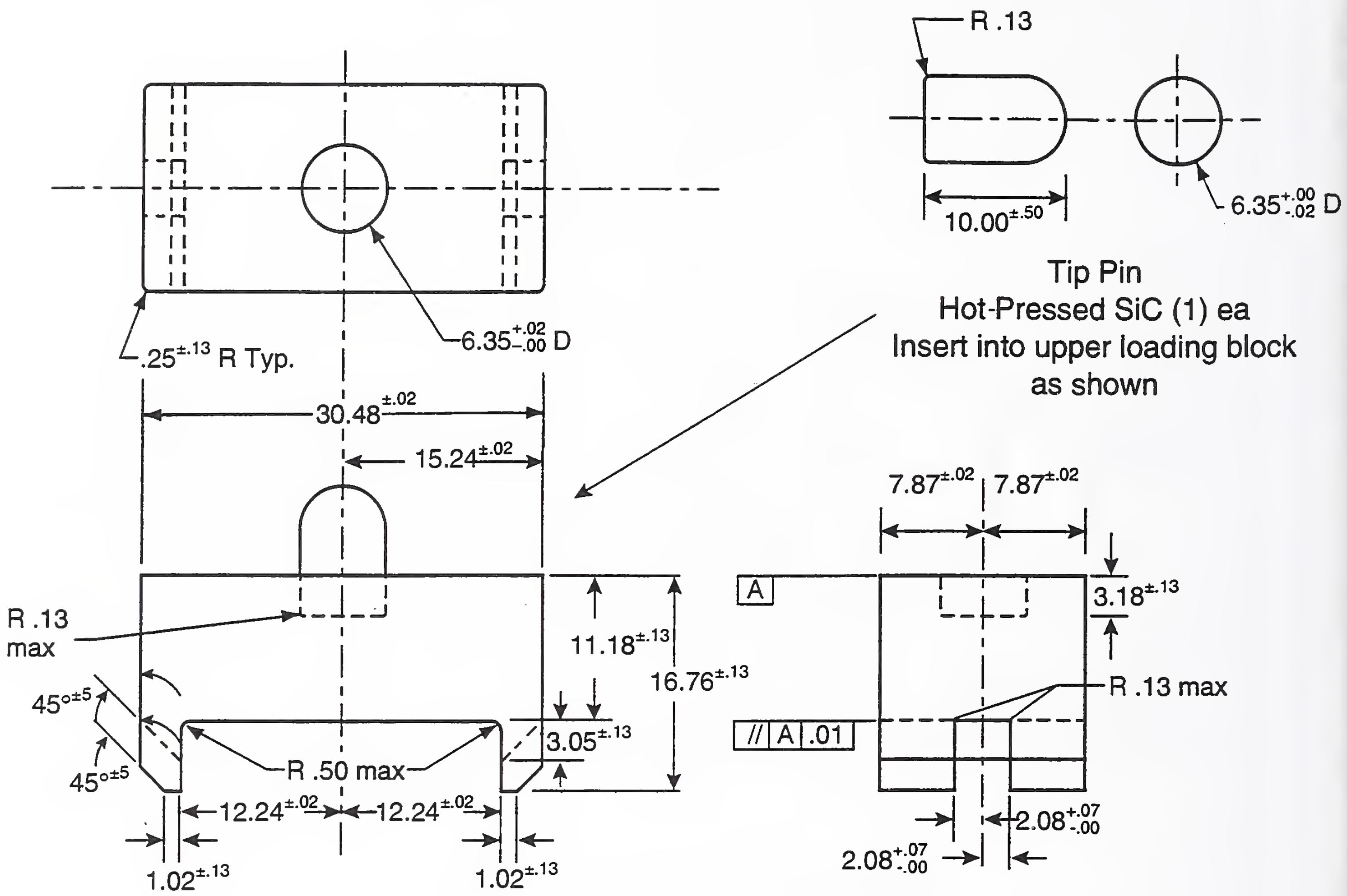
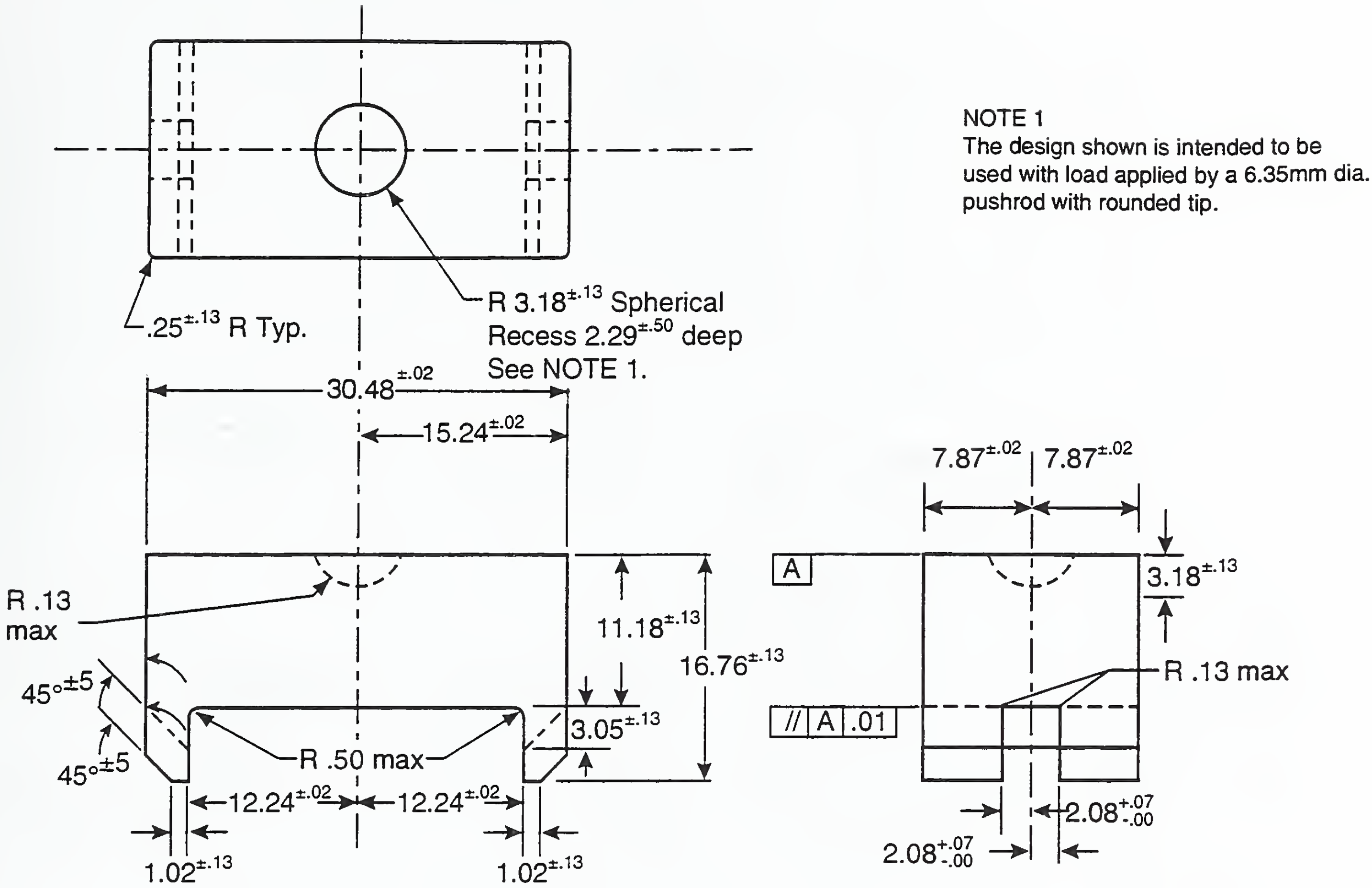


Figure 5. Guide Blocks. All dimensions in mm.



UPPER LOADING BLOCK
Sintered SiC (1) ea

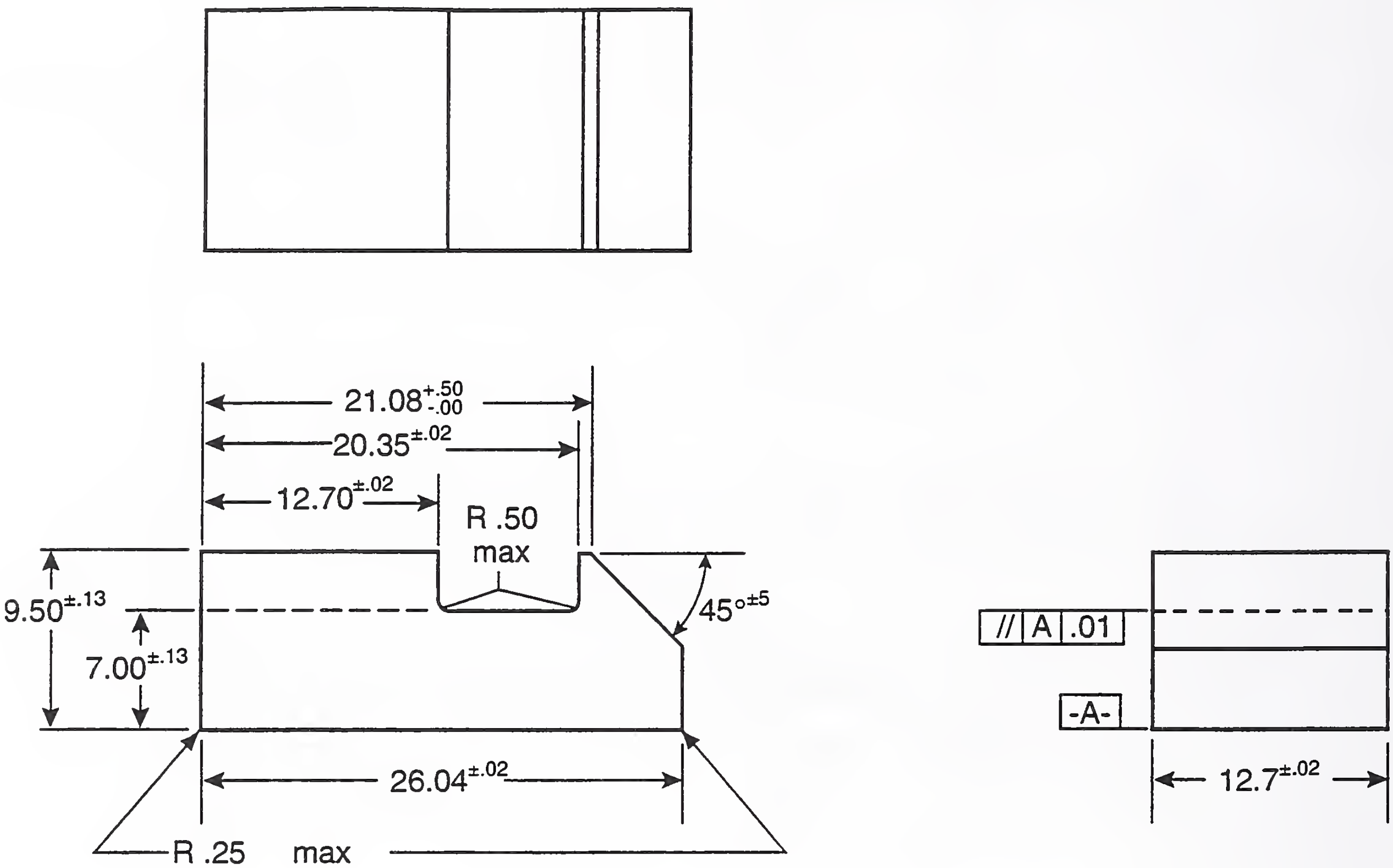
Figure 6. Upper Loading Block and Pin to be used with a flat-ended furnace pushrod. This piece can alternatively be made with hot-pressed silicon carbide. All dimensions in mm.



UPPER LOADING BLOCK - ALTERNATIVE

Sintered SiC (1) ea

Figure 7. An alternative Upper Loading Block for use with a rounded-tip furnace pushrod, or with a flat-tipped pushrod when with a ceramic ball bearing. This piece can alternatively be made of hot-pressed silicon carbide. All dimensions in mm.



LOWER ROLLER SUPPORTS
(2) ea Sintered SiC

Figure 8. Lower Roller Supports. All dimensions in mm.

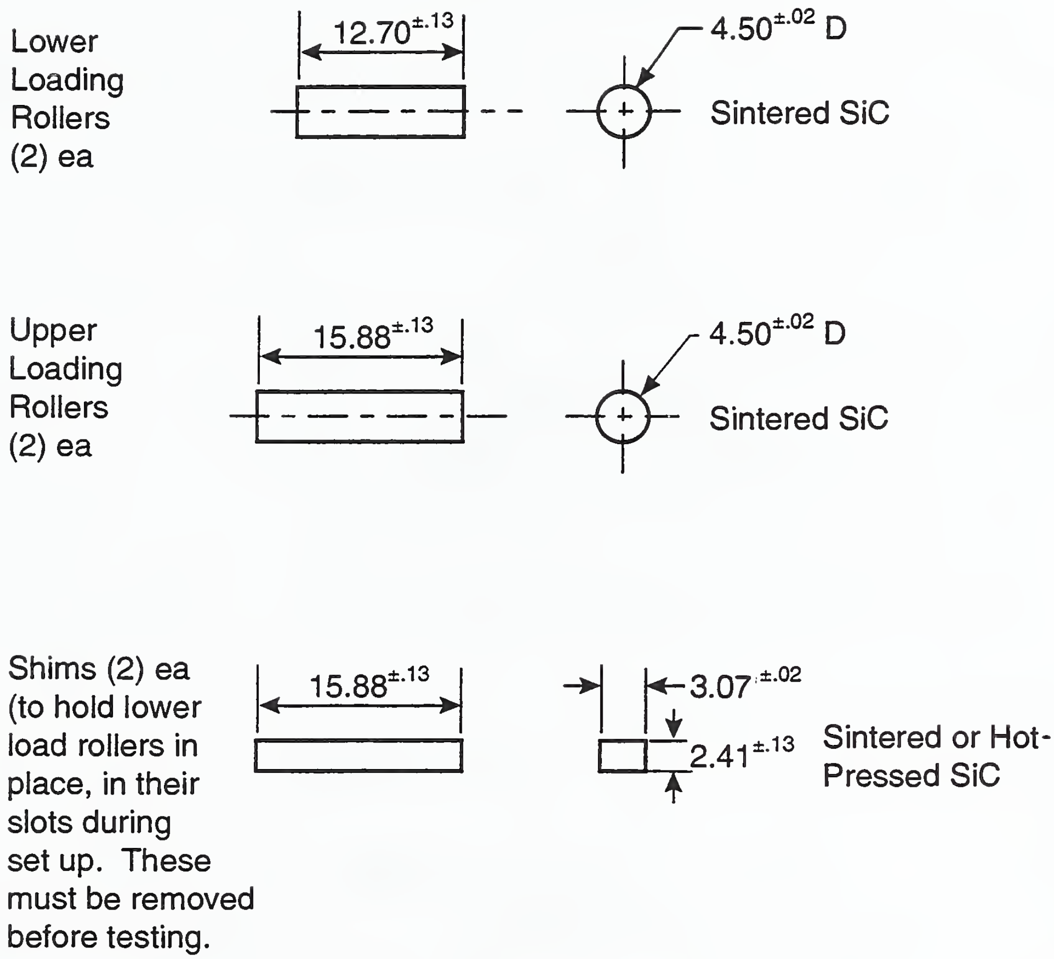


Figure 9. Loading Rollers and Shims. All dimensions in mm.

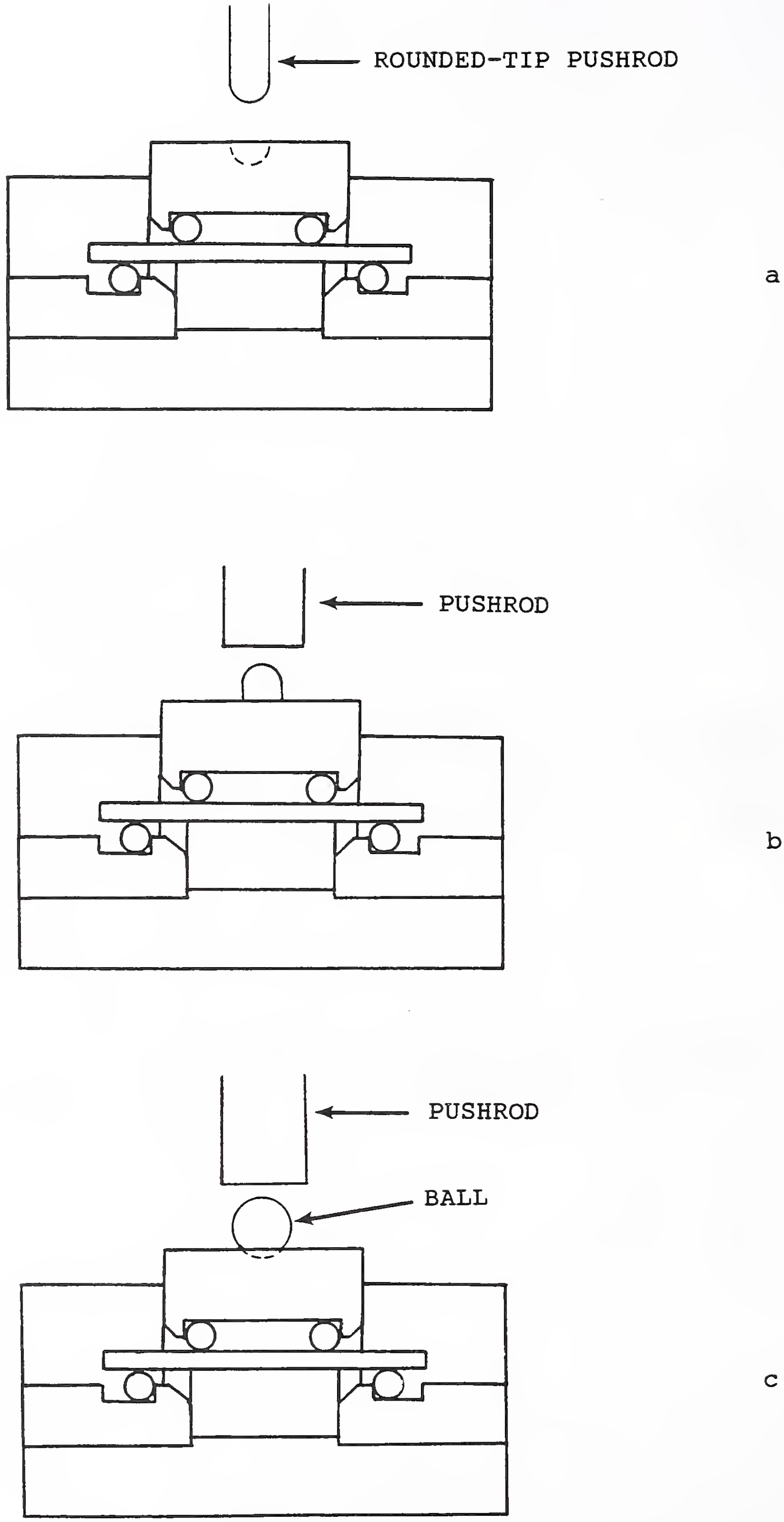


Figure 10. Possible loading arrangements.

APPENDIX

For convenience, the following drawings are furnished in non-SI units. The parts and their dimensions are identical to those shown in the text.

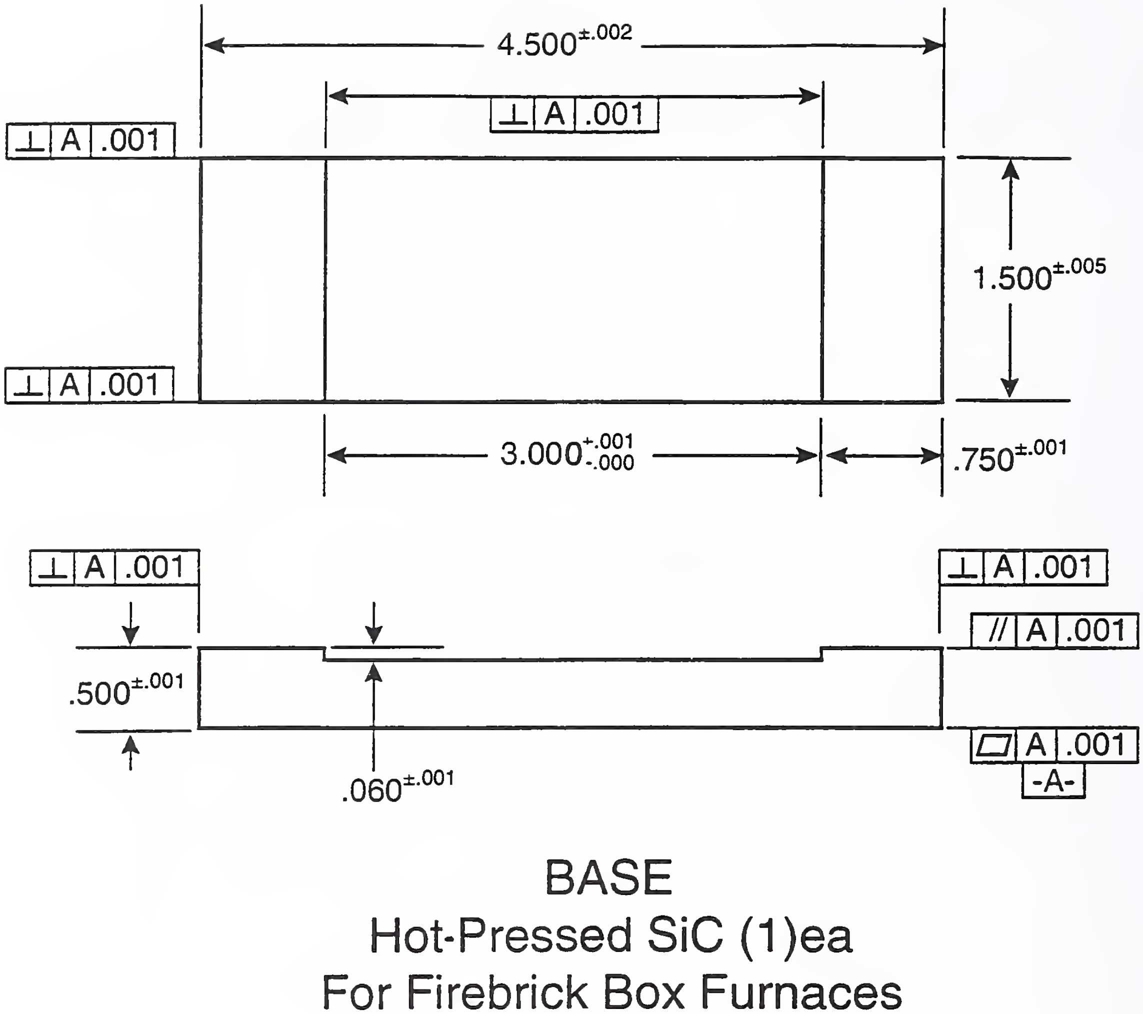
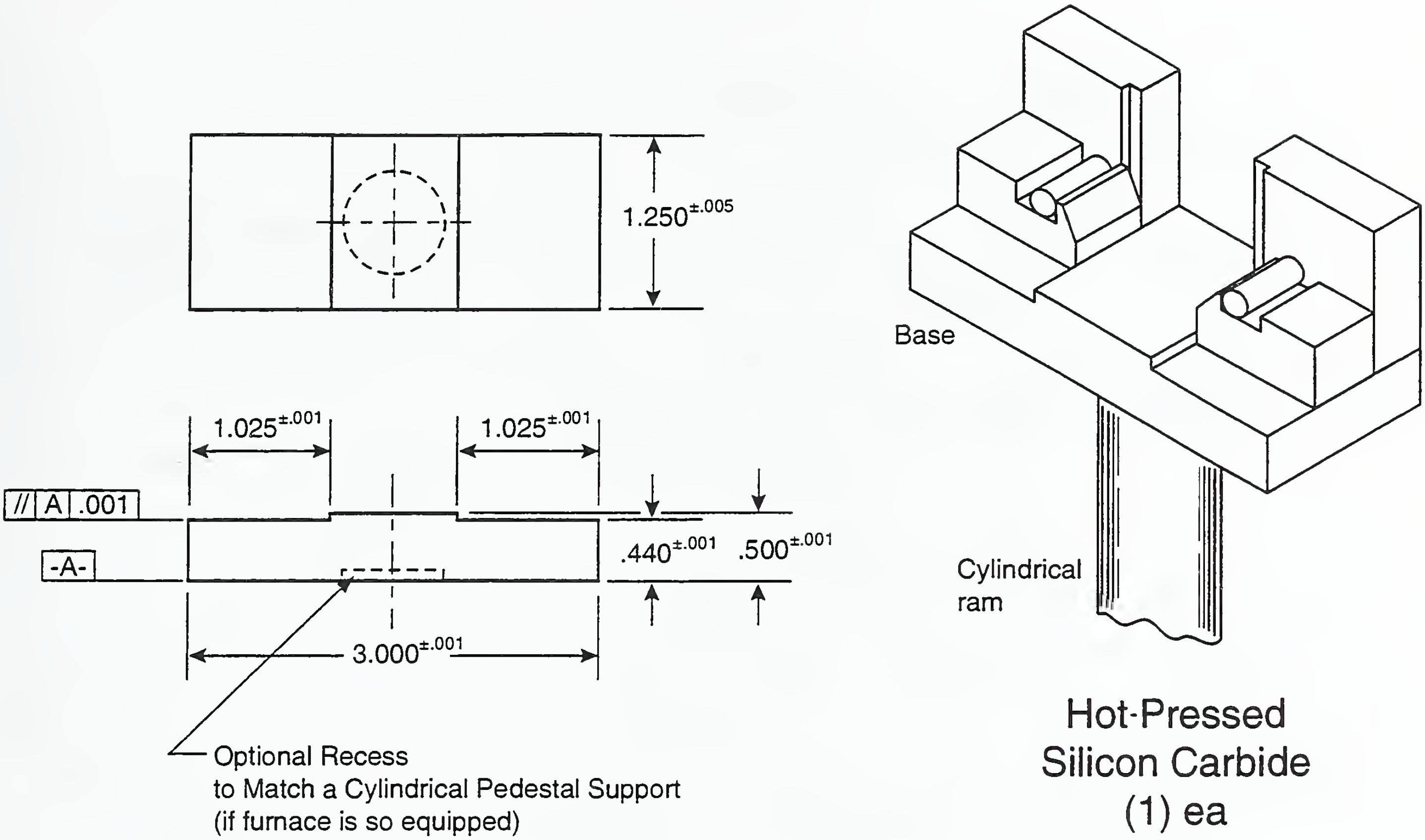


Figure A1. Base Plate. All dimensions in inches.



BASE - ALTERNATIVE STYLE
FOR CYLINDRICAL FURNACES OR FURNACES
WITH CYLINDRICAL RAMS OR PEDESTALS

Figure A2. Alternative Base Plate. All dimensions in inches.

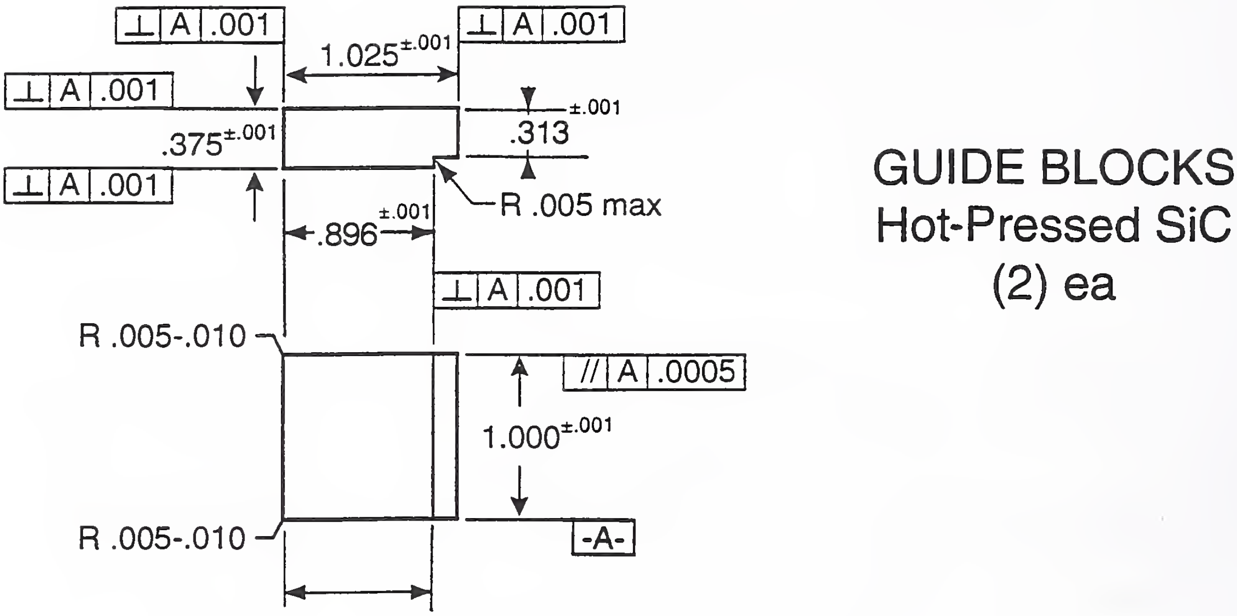
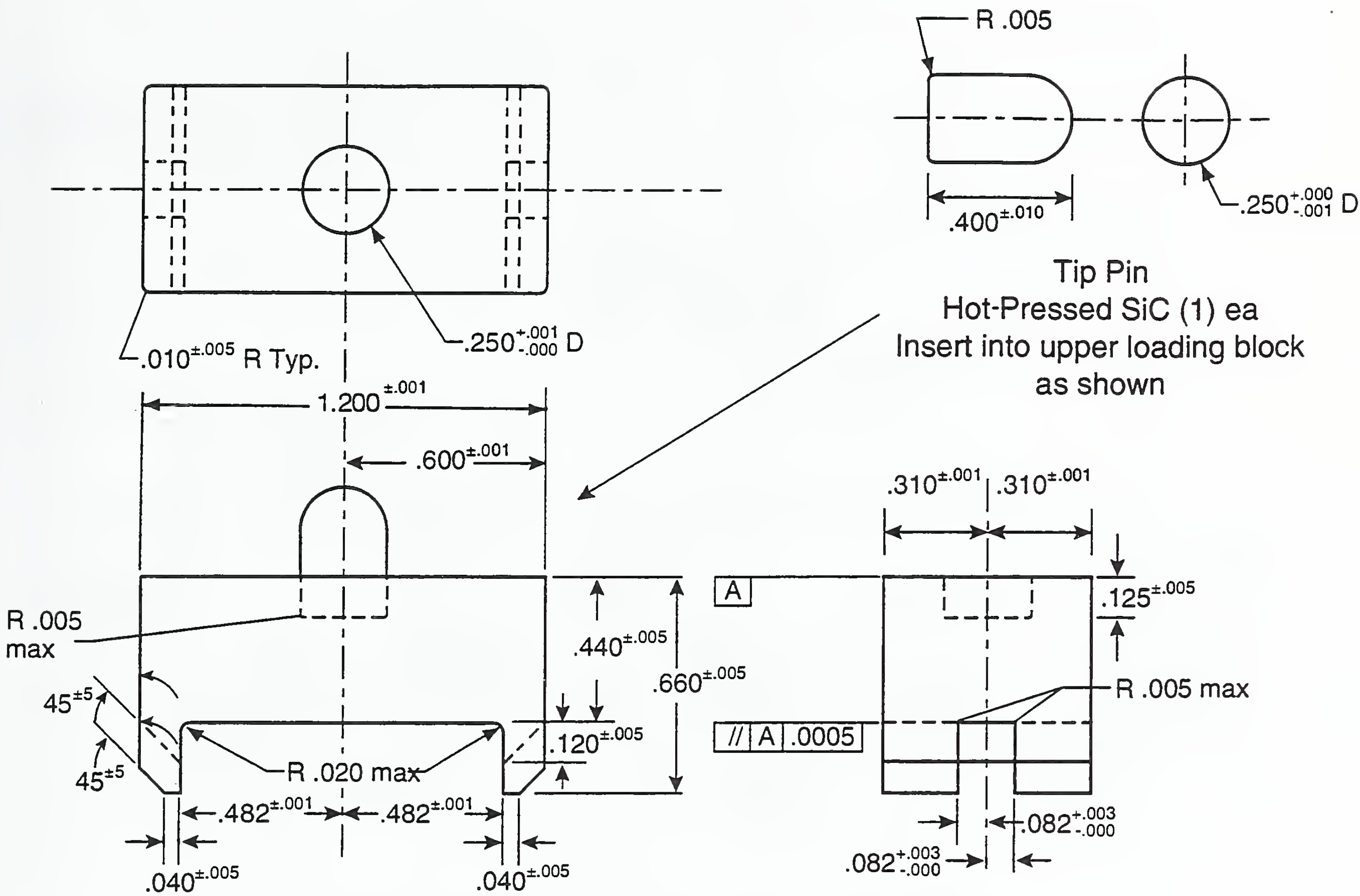
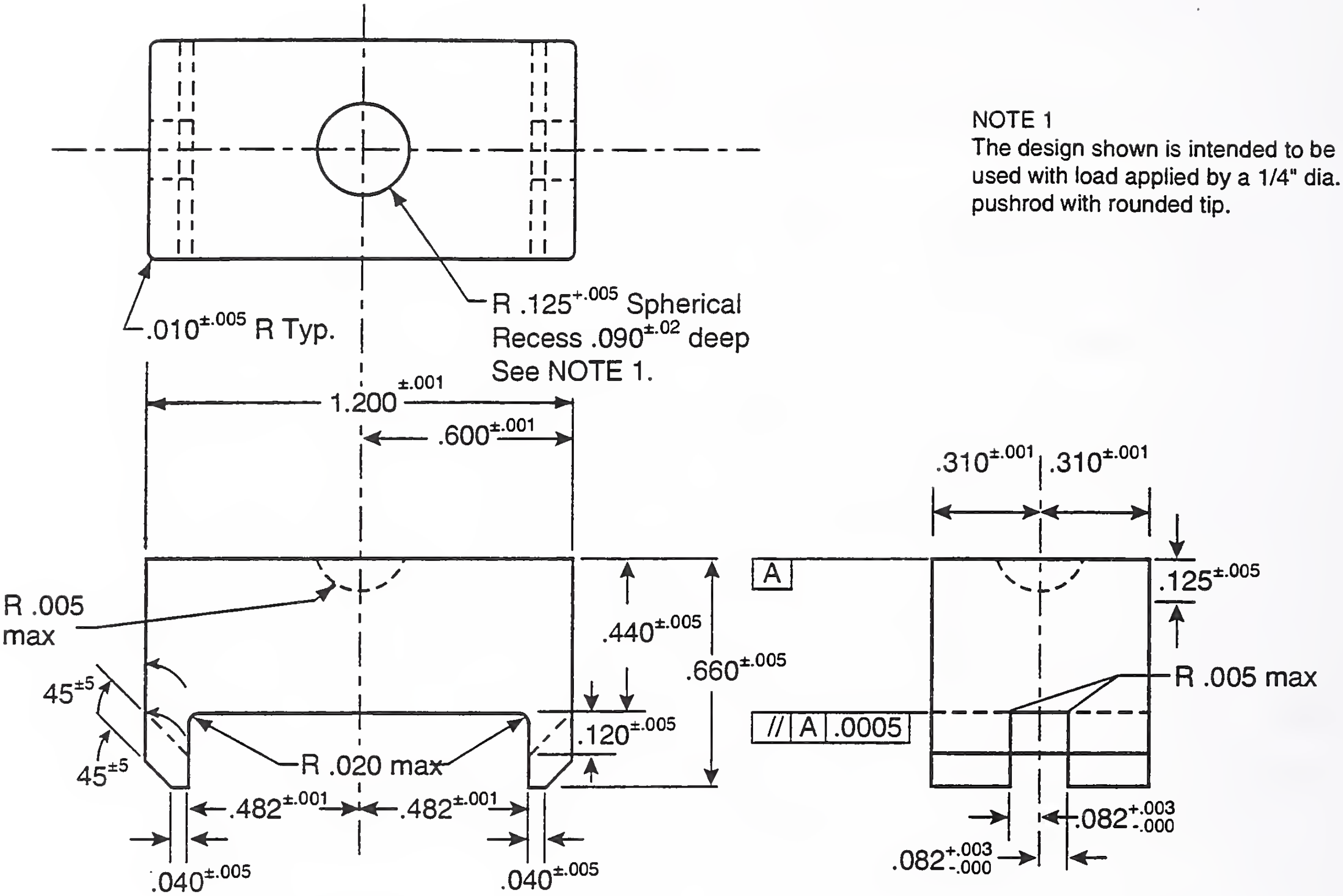


Figure A3. Guide Blocks. All dimensions in inches.



UPPER LOADING BLOCK
Sintered SiC (1) ea

Figure A4. Upper Loading Block. All dimensions in inches.



UPPER LOADING BLOCK - ALTERNATIVE
Sintered SiC (1) ea

Figure A5. Upper Loading Block, alternative design. All dimensions in inches.

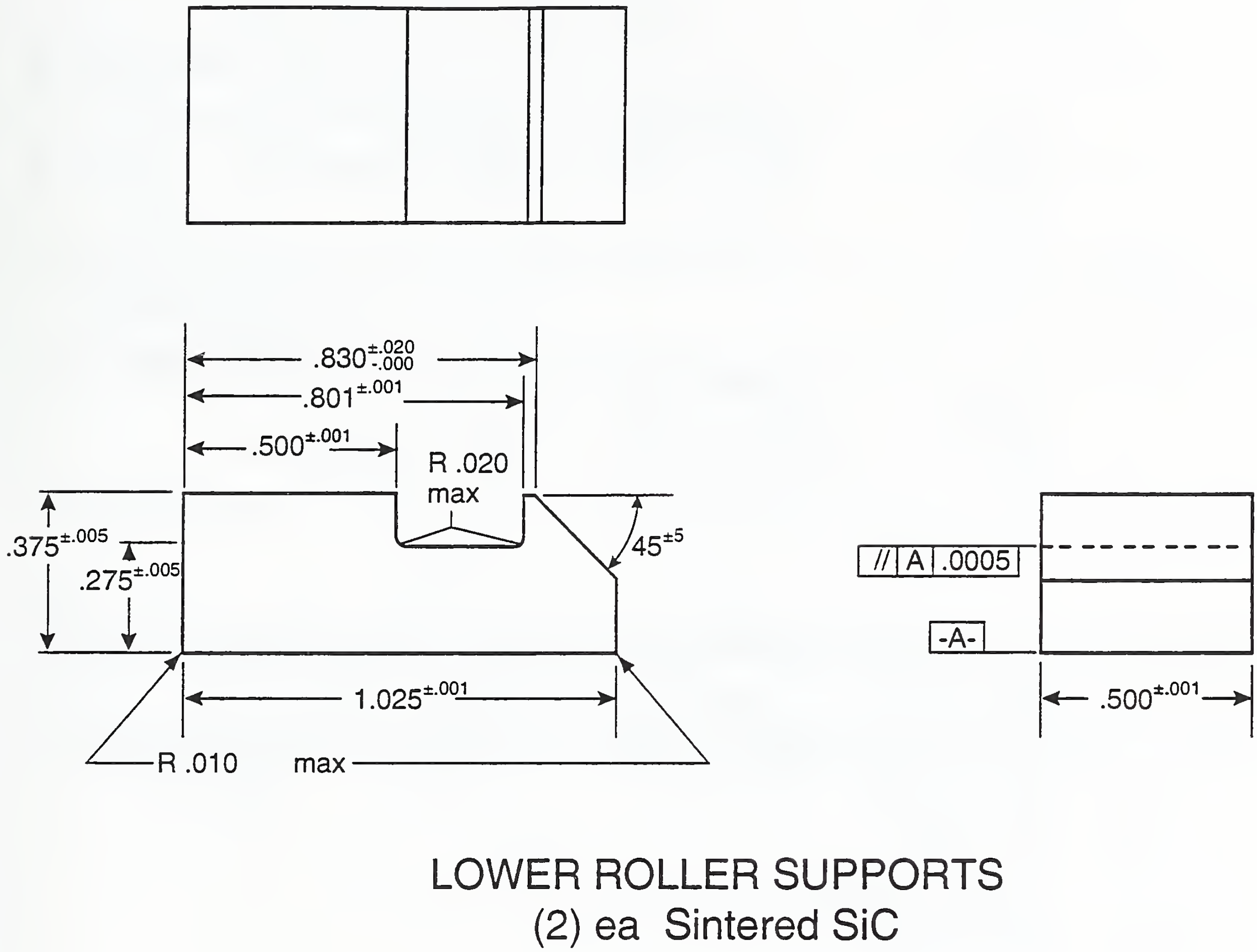


Figure A6. Lower Roller Supports. All dimensions in inches.

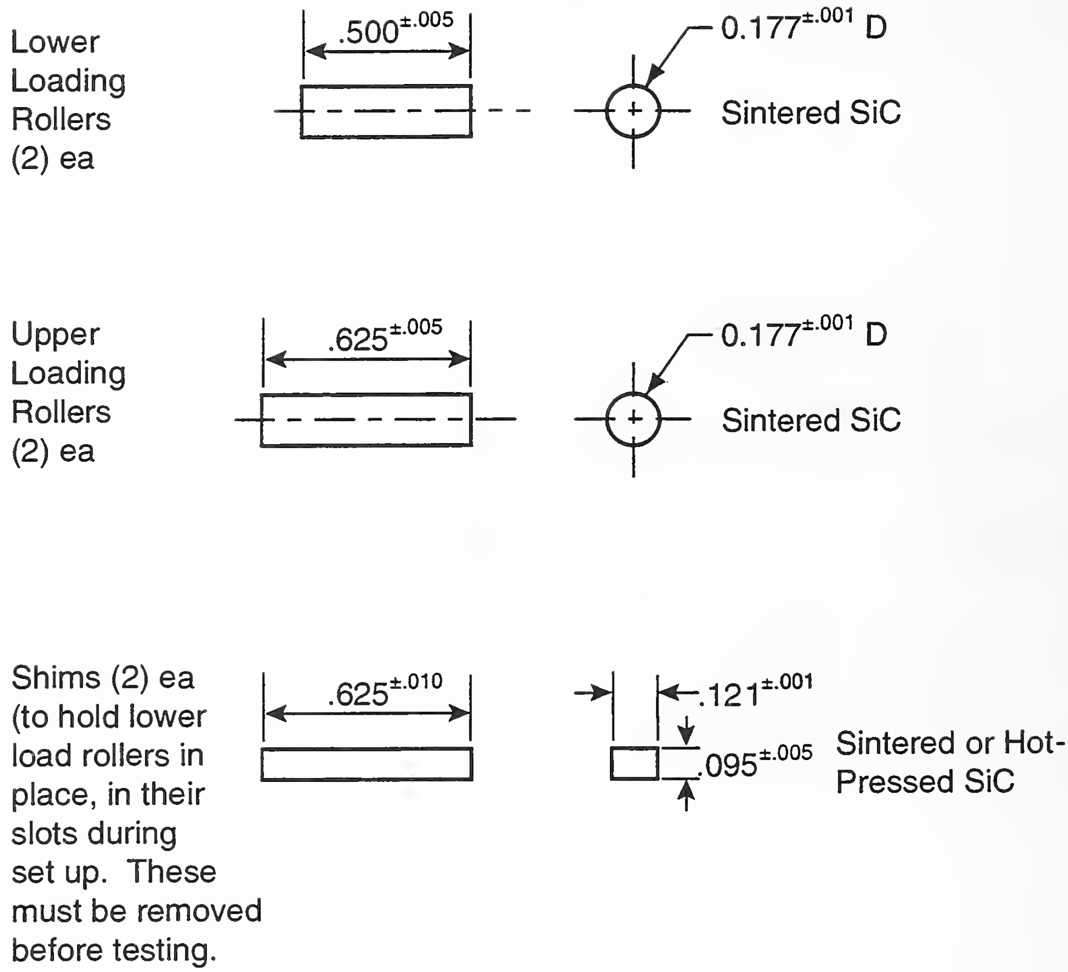


Figure A7. Loading Rollers and Shims. All dimensions in inches.

NIST-114A
(REV. 3-89)

U.S. DEPARTMENT OF COMMERCE
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY

BIBLIOGRAPHIC DATA SHEET

1. PUBLICATION OR REPORT NUMBER
NISTIR 4864

2. PERFORMING ORGANIZATION REPORT NUMBER

3. PUBLICATION DATE
JUNE 1992

4. TITLE AND SUBTITLE
HIGH-TEMPERATURE FLEXURE FIXTURE FOR ADVANCED CERAMICS

5. AUTHOR(S)
George Quinn

6. PERFORMING ORGANIZATION (IF JOINT OR OTHER THAN NIST, SEE INSTRUCTIONS)
U.S. DEPARTMENT OF COMMERCE
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
GAITHERSBURG, MD 20899

7. CONTRACT/GRANT NUMBER

8. TYPE OF REPORT AND PERIOD COVERED

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (STREET, CITY, STATE, ZIP)

10. SUPPLEMENTARY NOTES

[ ] DOCUMENT DESCRIBES A COMPUTER PROGRAM; SF-185, FIPS SOFTWARE SUMMARY, IS ATTACHED.

11. ABSTRACT (A 200-WORD OR LESS FACTUAL SUMMARY OF MOST SIGNIFICANT INFORMATION. IF DOCUMENT INCLUDES A SIGNIFICANT BIBLIOGRAPHY OR LITERATURE SURVEY, MENTION IT HERE.)

A test fixture has elevated temperature flexure strength testing is presented. The fixture is suitable for fast fracture or stress rupture experiments up to 1500 C in air or inert environments.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)
advanced ceramics; ASTM C1211; fixture; flexure strength; friction; high-temperature; silicon carbide

13. AVAILABILITY

[X] UNLIMITED

[ ] FOR OFFICIAL DISTRIBUTION. DO NOT RELEASE TO NATIONAL TECHNICAL INFORMATION SERVICE (NTIS).

[ ] ORDER FROM SUPERINTENDENT OF DOCUMENTS, U.S. GOVERNMENT PRINTING OFFICE, WASHINGTON, DC 20402.

[X] ORDER FROM NATIONAL TECHNICAL INFORMATION SERVICE (NTIS), SPRINGFIELD, VA 22161.

14. NUMBER OF PRINTED PAGES
25

15. PRICE
A02

ELECTRONIC FORM