NASA CR-111948

TENSILE MICROSTRAIN PROPERTIES OF TELESCOPE MIRROR MATERIALS

Final Report

June 1971

Prepared under Contract NAS1-9982 by

STANFORD RESEARCH INSTITUTE
Menlo Park, California 94025

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By John W. Moberly
Stanford Research Institute

SUMMARY

Fused silica, ULE fused silica, and Cer-Vit are nonmetallic mirror materials being considered for use in diffraction-limited optical systems. A critical property of materials for these systems is a high microyield stress. Microstrain tensile tests were made to measure the microyielding behavior of the three materials and these are compared to an alloy of beryllium plus 5 wt% copper. The strain sensitivity for the tests was 5×10^{-8}.

It was found that fused silica, ULE fused silica, and Cer-Vit, having etched surfaces behaved in an elastic manner showing no microyielding. Tests on fused silica and Cer-Vit having unetched surfaces produced similar results, in that no microyielding was observed. The samples were stressed to 34 to 38 MN/m^2 (5000 to 5500 psi) or to their fracture point if it occurred at a lower stress. The beryllium-copper alloy showed a permanent strain after loading to above 21 MN/m^2 (3000 psi).

The results on the etched nonmetallic samples agree with results determined by previous studies. However, the behavior observed for the unetched samples does not agree with the earlier results, which showed microyielding.

INTRODUCTION

The suitability of a material for use as a mirror substrate in a high precision optical system is based on several criteria. These include the dimensional stability, the quality of optical surface that can be obtained, and the strength of the material with regard to its use in optical systems. Strength, here, refers to the stress level to which the material may be subjected without its being permanently (plastically) deformed. Diffraction-limited mirrors should have a strain stability of better than 10^{-7}, approaching 10^{-8}; the ideal material would be perfectly elastic. The objective of this program was to determine the tensile microstrain characteristics of fused silica, ULE (ultra-low-expansion) fused silica, and Cer-Vit. The tests were to have a strain sensitivity of 5×10^{-8}. Both as-ground (unetched) and etched specimens were to be tested to determine how the surfaces influence the microstrain behavior.

A critical influence on the mechanical and micromechanical behavior of a material is its prior thermomechanical history. Surface effects have recently been observed in studies on the three mirror materials (Ref. 1,2). As-ground fused silica, ULE fused silica, and Cer-Vit showed microyielding at low stress levels when tested with the surfaces in a ground condition. However, chemically etching the surfaces to remove the damaged layer caused by grinding produced specimens that exhibited no microyielding at the same stress. These measurements were made using a strain sensitivity of 10^{-8}. The tests were made using tension, compression, and torsion loading. The earlier results were to be confirmed, if possible, with the tests conducted in this program.

All measurements of stress in the present study were in English units (psi). This report is written to conform to NASA specifications using units recommended by the International Union of Pure and Applied Physics -the International System units (meganewtons per square meter).

Since these units are unfamiliar to many readers, the English units are included in parentheses in this text.

EXPERIMENTAL PROCEDURES

Microstrain Test Equipment

The testing procedure used here for measuring microstrain is basically a hoop-stress technique with hydrostatic loading applied to the inside surface of a ring-shaped specimen through a thin elastomeric membrane (Ref. 3,4). The applied pressure is therefore always perpendicular to the specimen surface and is perfectly uniform over the entire ring periphery. The specimen to be tested is unconstrained (free-floating) during the test. There are, therefore, no clamps or holders to cause misalignment or localized stress concentrations. A diagram of the testing head is shown in Figure 1.

The basic testing head is a two-section stainless steel assembly. A porous thin metal shell is inserted over the base of the test head and a rubber bag is then stretched over the shell. At the ends the rubber is tucked beneath the shell, and a leak-proof seal is accomplished by two O-rings that are placed between the metal shell and the testing head. A cap is then positioned over the end of the test head and rigidly held by a pin, as shown in the drawing. Hydraulic fluid (in this case, water) enters the assembly at the end of the test head and passes through the thin-wall metal shell to inflate the rubber bag.

The test specimens are right circular rings 55.88 mm (2.200 in.) o.d., 50.80 mm (2.00 in.) i.d., and 3.35 mm (0.250 in.) high. To eliminate any friction between the specimen and the testing head, a small gap is left at the ends of the specimen. This gap is kept small to prevent the rubber from protruding and thereby applying a load to the ends of the specimen.

A capacitance system is used to measure changes in ring diameters. The specimen and assembled capacitor plates are shown in Figure 2. Small tabs are fastened diametrically on the ring with epoxy adhesive. Into one tab is screwed a flat 28.6 mm (1.125 in.) diameter stainless steel capacitor plate. A yoke assembly holding a second stainless steel plate, diameter 25.4 mm (1 in.), is then attached to the other

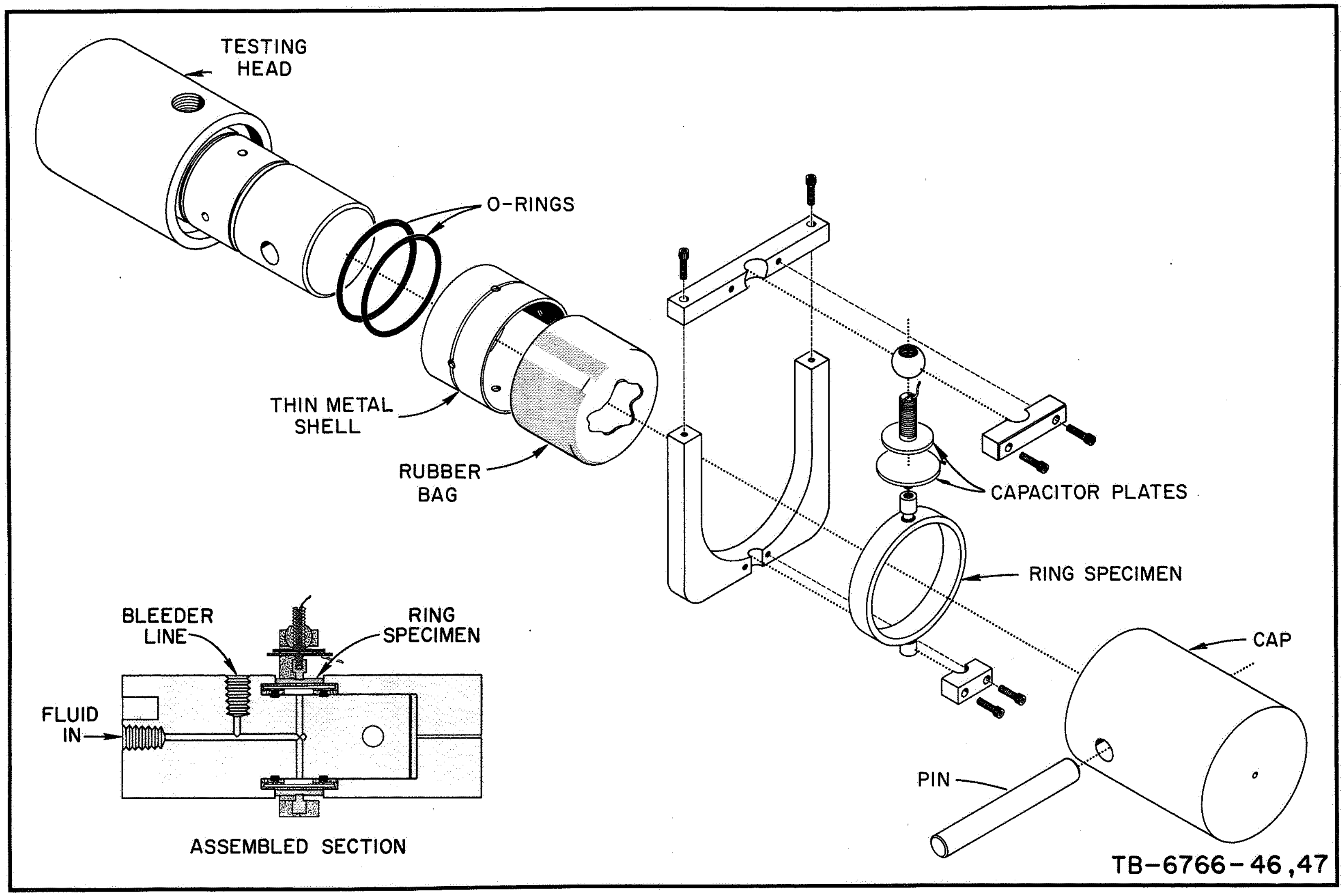

FIGURE 1 SCHEMATIC SKETCH OF MICROSTRAIN TEST APPARATUS

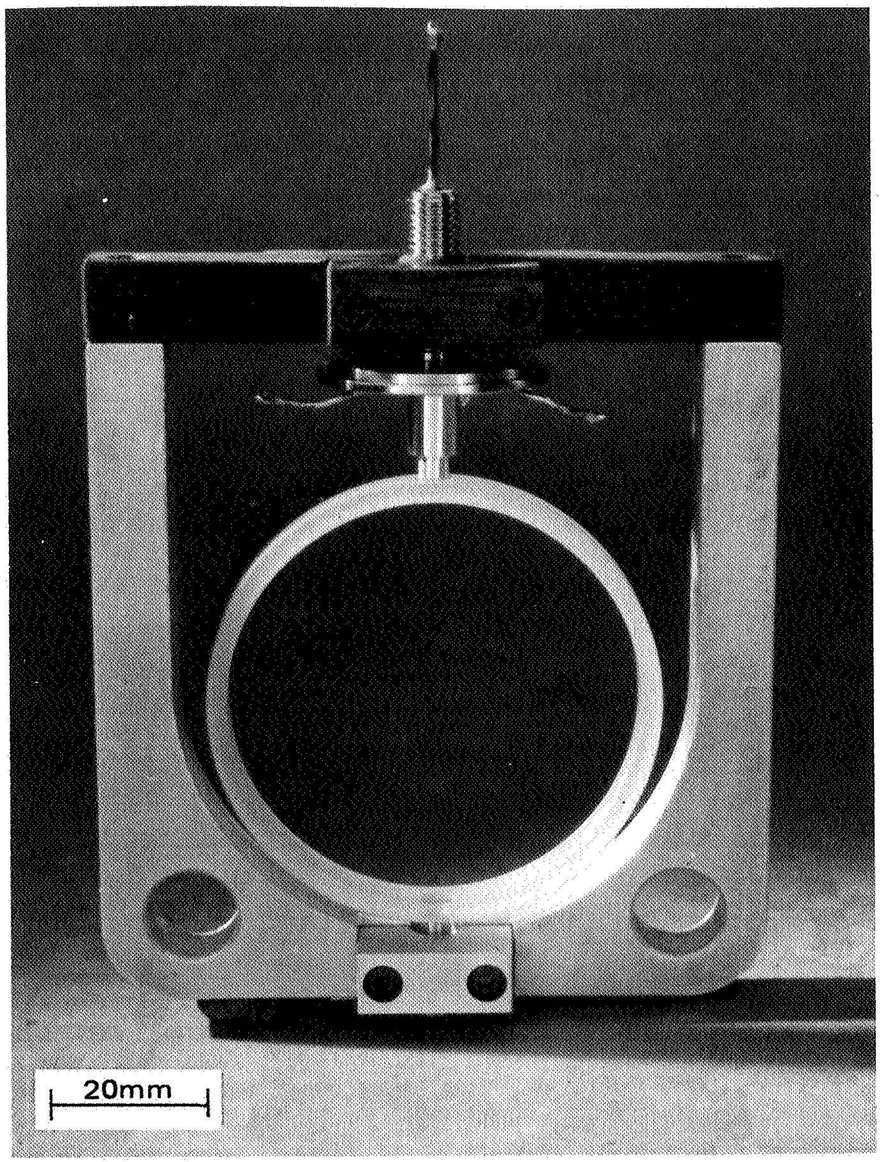

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FIGURE 2 TEST SPECIMEN WITH CAPACITANCE GAUGES tab. The plates can be aligned parallel by the ball adjustment at the top of the yoke. The plates are spaced about 0.10 mm (0.004 in.) so as to produce an interplate capacitance of about 30 picofarads.

The capacitor-transducer is calibrated to determine the strain sensitivity by attaching similar capacitor plates to a Fabry-Perot interferometer and determining the distance-capacitance relationship. The sensitivity is then checked during each test by relating the Young's modulus of the test sample to the applied microstrain stress and calculating the strain. Correlation of the strains by the two techniques has been excellent and allows for detection of strains of less than 5 x 10^{-8}. This represents a voltage output signal from the capacitance transducers of about 2 mV per 10^{-6} strain. At this sensitivity, the signal-to-noise ratio is approximately 50:1.

The stress on the test specimen is determined by recording the applied hydrostatic force on the sample and calculating the stress from elasticity equations. The resolved stresses within the ring are defined mathematically by

$$\sigma_t = \frac{Pr_i^2}{(r_o^2 - r_i^2)} \left(1 + \frac{r_o^2}{r^2}\right) \qquad (1)$$

$$\sigma_r = \frac{Pr_i^2}{(r_o^2 - r_i^2)} \left(1 - \frac{r_o^2}{r^2}\right) \qquad (2)$$

where

σ_t = tangential tensile stress

σ_r = radial compressive stress

P = hydrostatic pressure

r = specimen radius at σ_t or σ_r

r_i = internal specimen radius

r_o = external specimen radius.

The tensile strain is given by the ratio

$$\epsilon = \frac{\Delta r}{r} = \frac{\Delta d}{d} \tag{3}$$

During a test, the assembly is contained in a double-walled box to minimize temperature changes. Because of the extremely high strain sensitivity, it is possible to detect the small length changes caused by thermal expansion and contraction of the test sample and strain detection fixture, even though the temperature fluctuation during a test is usually less than 0.1°C. However, these changes as well as any electronic changes, are observed as continuous drift.

The entire test assembly is placed on a granite block that is floated on air pillows. Thus the measurements are not influenced by vibrations.

About four hours is sufficient for temperature stabilization before a test. The specimen is preloaded to 0.69 MN/m^2 (100 psi) to accommodate any minor displacement in the loading mechanism. This stress then becomes the reference level for all further measurements. The peripheral tensile stress is calculated from hydraulic line pressures according to Eq. (1).

The hydrostatic pressure is measured by a dial gauge calibrated to 0.5%. The test sample is loaded and unloaded in increments of 3.4 MN/m^2 (500 psi). Loads are held for 30 seconds. The sample is then unloaded to 0.69 MN/m^2 (100 psi). Any permanent change in diameter is recorded as residual plastic strain; the tensile strain is obtained by Eq. (3).

Test Specimens

The three materials evaluated were:

(1) Fused silica - Corning Glass fused silica No. 7940 (mirror blank quality).

(2) ULE Fused silica - Corning Glass fused silica No. 7971 (mirror blank quality).

(3) Cer-Vit - Owens Illinois Cer-Vit No. C-101 (premium grade mirror blank).

Fused silica is high purity silicon dioxide having a glass structure. ULE fused silica is also a silica glass but with the addition of 7% titanium dioxide. The effect of this addition is a shift of the region of nearly zero thermal expansion from -200°C (as for fused silica) to ambient temperatures.

Cer-Vit is a devitrified lithium-aluminum silicate containing approximately 10% glass phase. The grain size is extremely fine, less than one micrometer. Cer-Vit, like ULE fused silica, has a zero coefficient of thermal expansion at ambient temperatures.

The samples of each material were obtained from their respective manufacturers. The Cer-Vit ring specimens were received in the final test dimensions. The fused silica and ULE fused silica samples were obtained oversize, and final grinding was done at a commercial ceramic grinding laboratory.

In addition, an isotropic alloy of beryllium plus 5 wt% copper, fabricated at SRI, was evaluated. The purpose was to compare the microstrain behavior of a metal with that of the three nonmetallic mirror materials.

Test Procedure

NASA specifications required that the samples be ground on all surfaces to a surface finish of at least 0.4 μm (16 μin.) rms. The Cer-Vit rings were very good quality in that there were no visible chips or cracks on any surface. The surface finish as measured with a surface profilometer was 0.33 to 0.40 μm (16 μin.) rms. The fused silica and ULE fused silica samples after final grinding were not as good as the Cer-Vit samples in one respect--there were small cracks and chips on the surfaces, generally at the edges. However, the surface finish--0.13 to 0.15 μm (5 to 6 μin.) rms--was somewhat better than that on the Cer-Vit samples.

The ground rings were stress relieved by annealing in air at 525°C for one hour and then slowly cooling to room temperature.

Two samples each of the fused silica, ULE fused silica, and Cer-Vit were etched to remove 0.10 to 0.12 mm (0.004 to 0.005 in.) from all surfaces. For fused silica and ULE fused silica the etchant was a 48% aqueous solution of hydrofluoric acid. For the Cer-Vit samples the hydrofluoric acid was diluted to 24%. Figure 3 shows photomicrographs of the three mirror materials before and after etching.

In an initial series of experiments the microstrain behavior was somewhat erratic and the results from duplicate samples were not in good agreement, and large strains were detected at the points where the strain-detection capacitance plates were mounted with epoxy cement. These strains, detected by an optical birefringence technique, were introduced when the specimen was cooled from an epoxy curing temperature of 150°C. Since there must be corresponding strains in the epoxy, errors in the microstrain test results could be from strains in either the specimen or the epoxy, or both. The problem was corrected by using a four-day, room temperature cure for the epoxy. No strains were found in the ring samples when this slower curing procedure was used.

A second series of tests was then made using the room temperature cure. A sufficient number of Cer-Vit specimens remained for this repeat testing. However, additional specimens of fused silica and ULE fused silica had to be obtained from the manufacturer.

As before, the fused silica and ULE fused silica specimens were ground to final dimensions. In this series, to prevent breakage and chipping, the specimens were annealed at 525°C frequently during grinding. Even with these precautions only four fused silica and two ULE samples were produced. The surfaces were nearly completely free

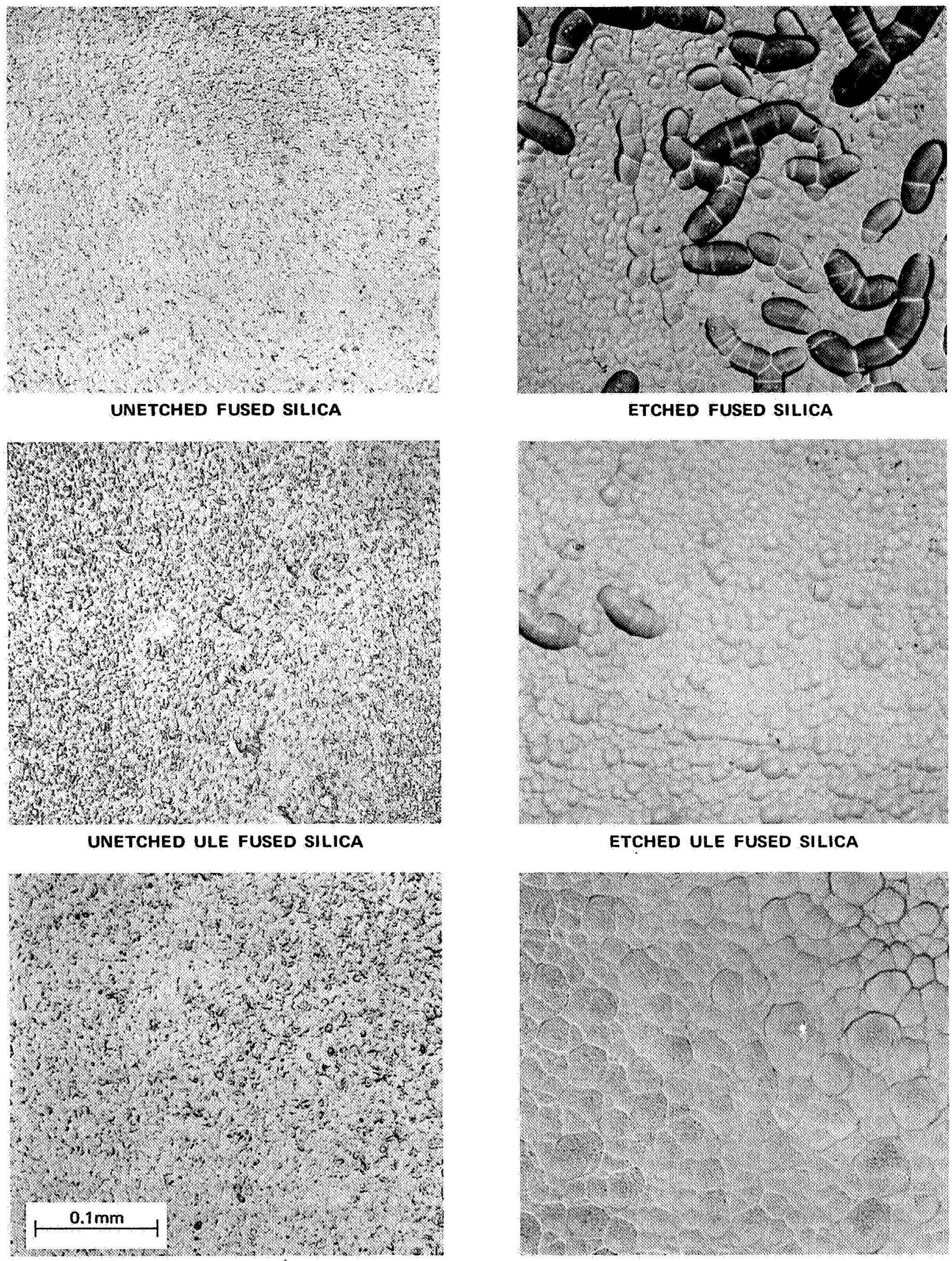

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FIGURE 3 PHOTOMICROGRAPHS OF THE THREE MIRROR MATERIALS BEFORE AND AFTER ETCHING of cracks and all had excellent surface finishes, about 0.13 μm (5 μin) rms. These and the Cer-Vit samples were given a final annealing at 525°C. Tests were made on Cer-Vit and fused silica in both etched and unetched conditions, but due to lack of samples data for ULE were only obtained in the etched condition.

RESULTS

One sample each of the three materials in the as-ground condition was to be tested to failure. The purpose of the fracture tests was to indicate the approximate limit of stress level to which samples could be loaded without being fractured. The intent was to preserve the specimens from subsequent strain tests for possible retesting. The ULE fused silica specimen cracked during mounting of the strain gauge for measurement of Young's modulus; therefore, only fused silica and Cer-Vit were tested to failure. The results of these two tests are:

Material	Fracture Strength	Young's Modulus
Fused silica	49 MN/m^2 (7000 psi)	71 MN/m^2 (10.3×10^6 psi)
Cer-Vit	76 MN/m^2 (11,000 psi)	81 MN/m^2 (12.6×10^6 psi)

These two specimens were representative of their respective classes in that the surface quality was good. However, these fracture strengths should not be taken as a true measure of the strength of the materials due to the possibility of microcracks and stress concentrations, which would cause premature fracture. Also, the fracture strengths here are the tangential stresses on the inner ring diameter, which is about 10% higher than the stress on the outer diameter--see Eq. (1). However, the Young's modulus values for both materials should be as meaningful as one can expect from single tests.

The microstrain test was based on stressing the samples to about 34 MN/m^2 (5000 psi). The stress reported for the microstrain tests is the tangential tensile stress on the outer ring diameter.

Fused Silica

The microstrain behavior for fused silica in the unetched (as-ground) and etched conditions is shown in Figures 4 and 5. No microyielding was observed in either of the two unetched samples. Sample FS-11 behaved elastically when stressed to 38 MN/m^2 (5500 psi). Sample FS-12 did show a strain of 5×10^{-8} (the detection limit) at 21 MN/m^2 (3000 psi) but did not yield further when loaded higher, until 34 MN/m^2 (5000 psi), at which point the sample fractured. Considering the total stress-residual strain curve it was concluded that this sample also was elastic to within the strain detection limit of the measuring system.

The results of etched fused silica are nearly identical to those of the unetched. No microyielding was observed when sample FS-14 was tested to 34 MN/m^2 (5000 psi). The second etched sample (FS-13) showed what appeared to be microyielding--a total strain of about 5×10^{-7} when loaded to the same stress level. This is the total strain measured and is the accumulated value from each of the previous stress levels. The onset of microyielding was at 17 MN/m^2 (2500 psi). In this test there was significantly more time-dependent (anelastic) strain recovery than in any of the previous tests. For sample FS-14 there was a total anelastic strain of about 10^{-6}, which appears to be fully recovered in about six to eight minutes. All but 10^{-7} was recovered in about four minutes. Sample FS-13, on the other hand, showed an anelastic recovery of over twice this amount, approximately 2.5×10^{-6} after being stressed to about 28 MN/m^2 (4000 psi), with a corresponding increase in time--over twelve minutes. This greatly increases the difficulty of comparing the initial base line of strain to that after loading, and effectively reduces the strain detection limit to about 2×10^{-7}. This raises an uncertainty as to whether the permanent strain measured for FS-13 was real. Considering the results of the other etched and unetched fused silica samples, it is concluded that no microyielding occurred.

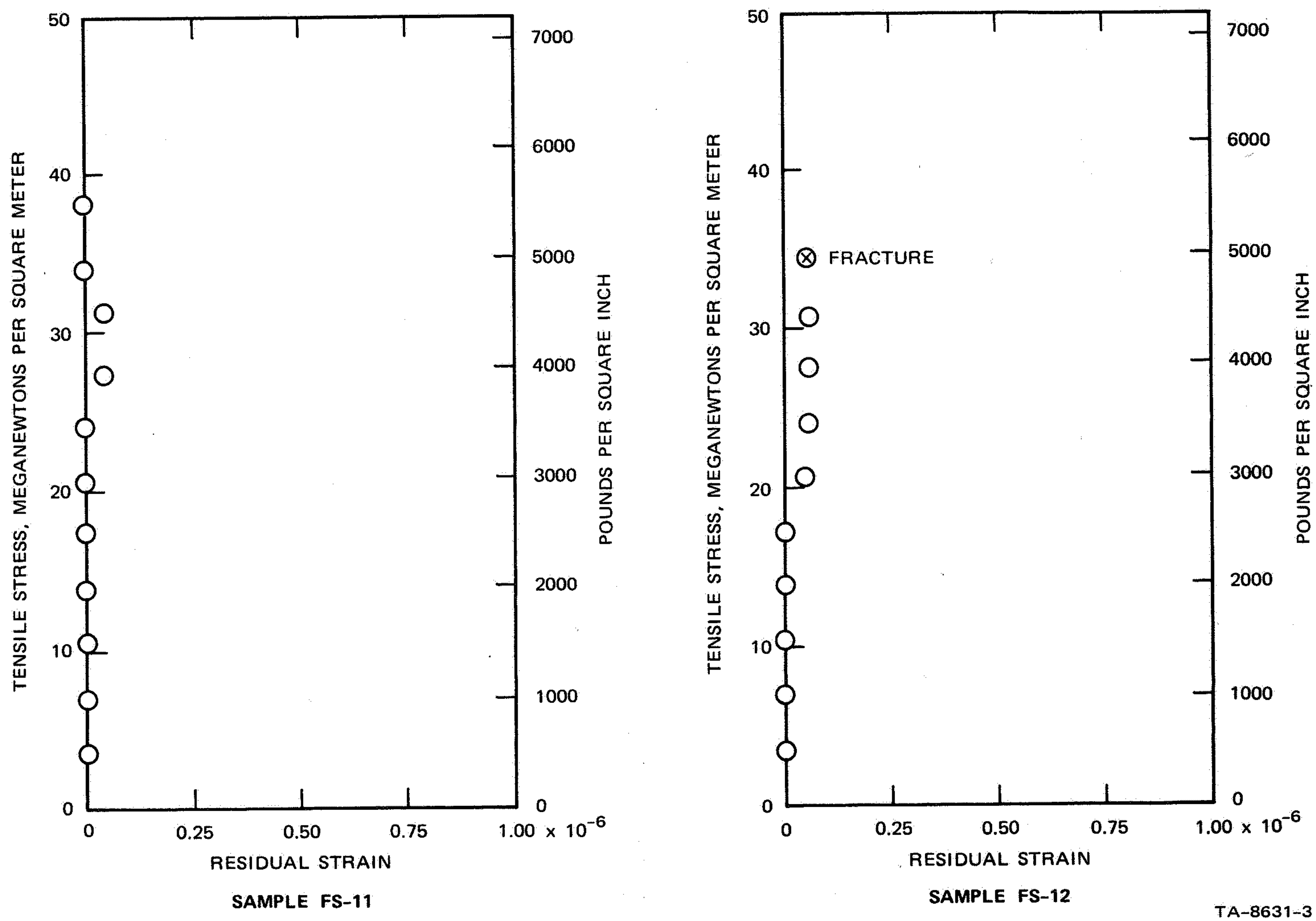

FIGURE 4 MICROSTRAIN MEASUREMENTS FOR TWO UNETCHED FUSED SILICA SPECIMENS

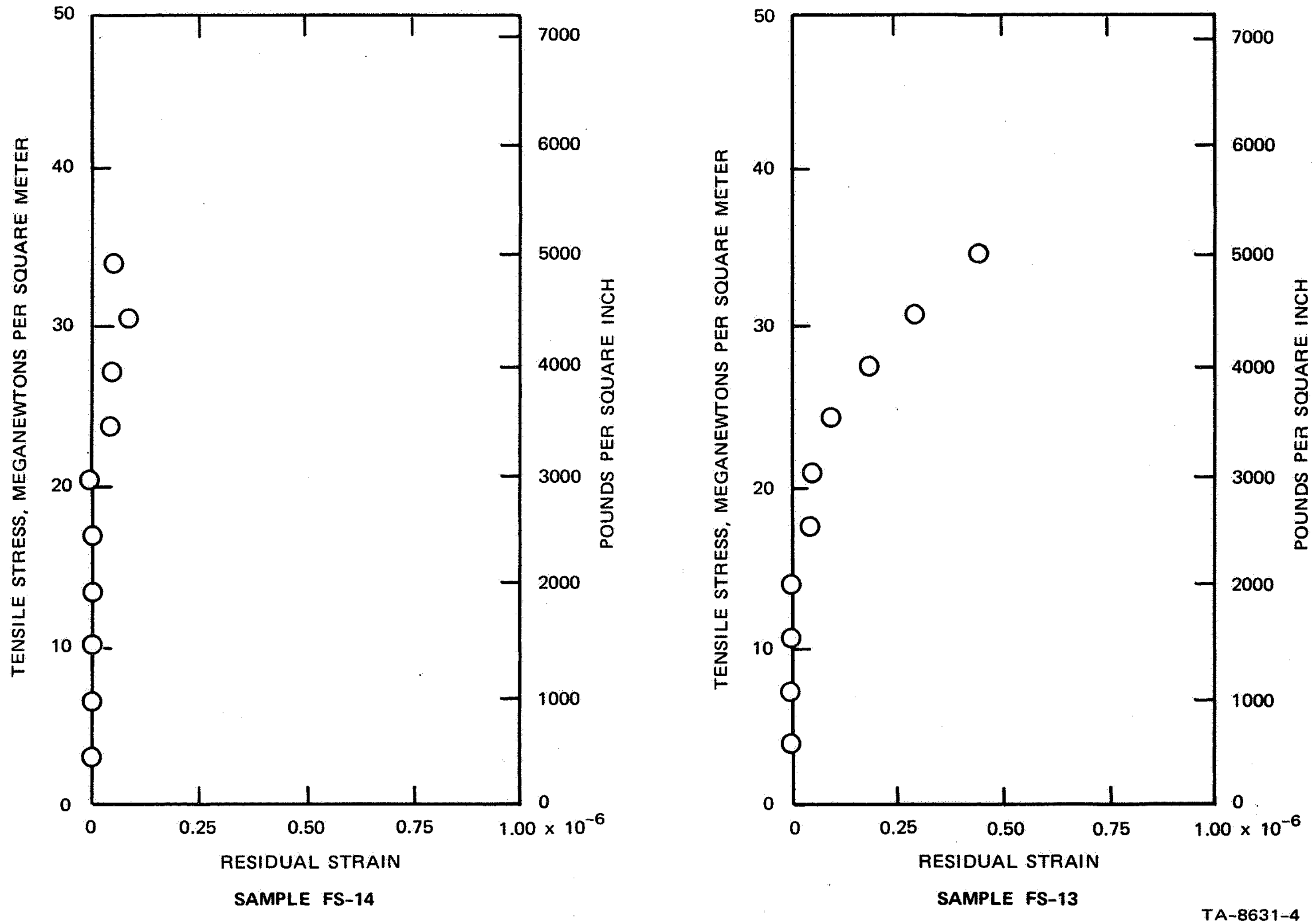

FIGURE 5 MICROSTRAIN MEASUREMENTS FOR TWO ETCHED FUSED SILICA SPECIMENS

ULE Fused Silica

Due to the fracture of specimens in final grinding the tests on ULE fused silica were limited to samples whose surfaces had been etched. The results are shown in Figure 6. Sample ULE-11 showed no microyielding when tested to 31 MN/m^2 (4500 psi). Sample ULE-6 also showed what is concluded to be zero microyielding until it fractured at 34 MN/m^2 (5000 psi). These results are effectively identical with the results from etched fused silica.

Cer-Vit

Etched and unetched Cer-Vit samples were also elastic. However, one unetched Cer-Vit sample failed at 34 MN/m^2 (5000 psi); the second, at 28 MN/m^2 (4000 psi). See Figures 7 and 8. This was somewhat surprising inasmuch as the initial fracture test specimen of this material (unetched) didn't break until 76 MN/m^2 (11,000 psi). This again shows that these fracture strengths are not indicative of the true fracture strength of the material, but more a measure of the surface quality (microcracks).

One of the etched Cer-Vit samples was loaded to a stress greater than 49 MN/m^2 (7000 psi). The sample did not fail and no microyielding was observed.

Beryllium-Copper Alloy

Two samples of an alloy of beryllium plus 5 wt% copper were tested. After fabrication and machining, the specimens were etched with a mixture of hydrofluoric and nitric acids to remove 0.10 mm (0.004 in.) from all surfaces. The results of the microstrain tests are shown in Figure 9. No microyielding was recorded for either specimen up to 21 MN/m^2 (3000 psi). A strain of 10^{-6} was then measured after loading to 31 to 34 MN/m^2 (4500 to 5000 psi). This behavior is characteristic of sintered beryllium.

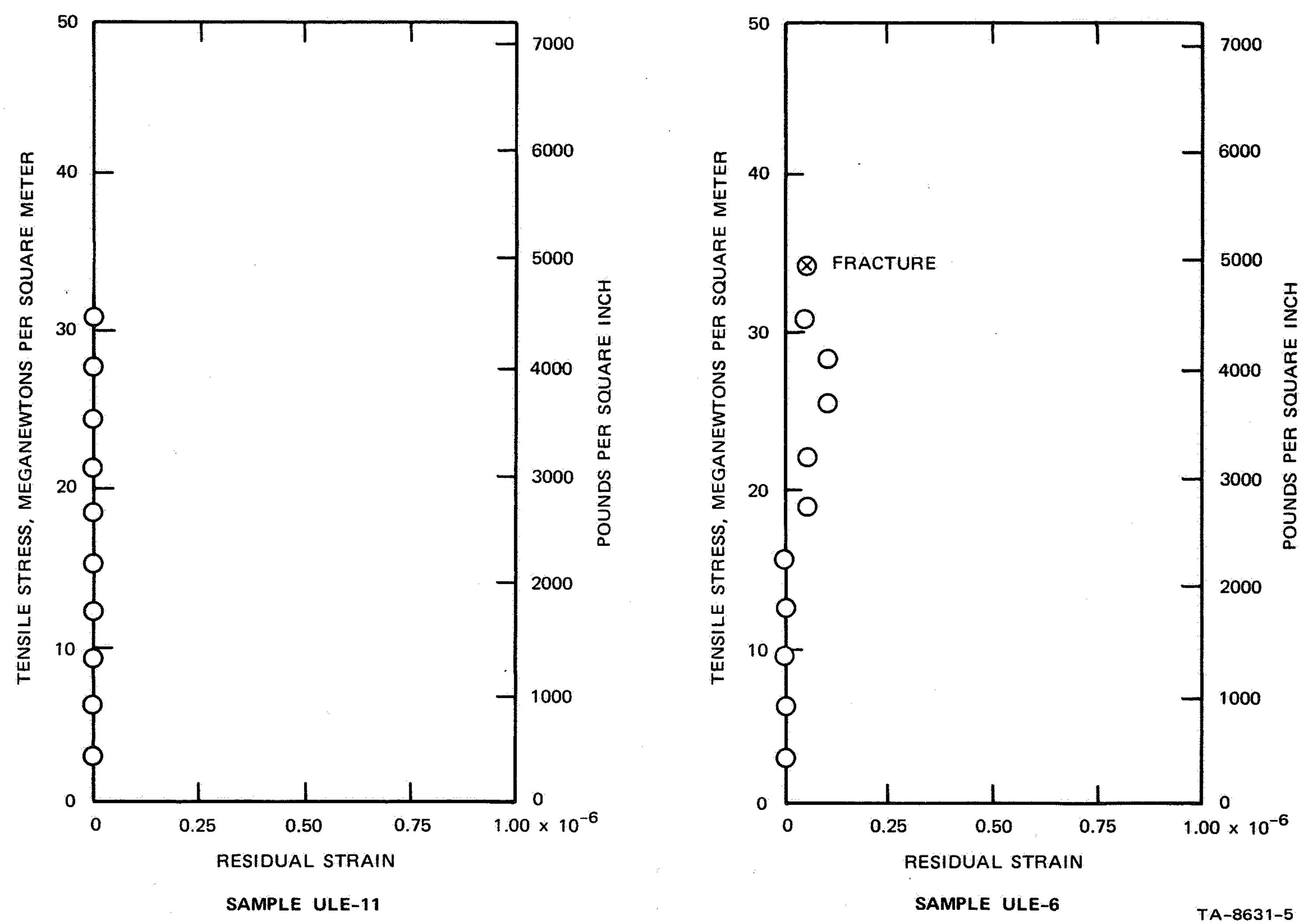

FIGURE 6 MICROSTRAIN MEASUREMENTS FOR TWO ETCHED ULE FUSED SILICA SPECIMENS

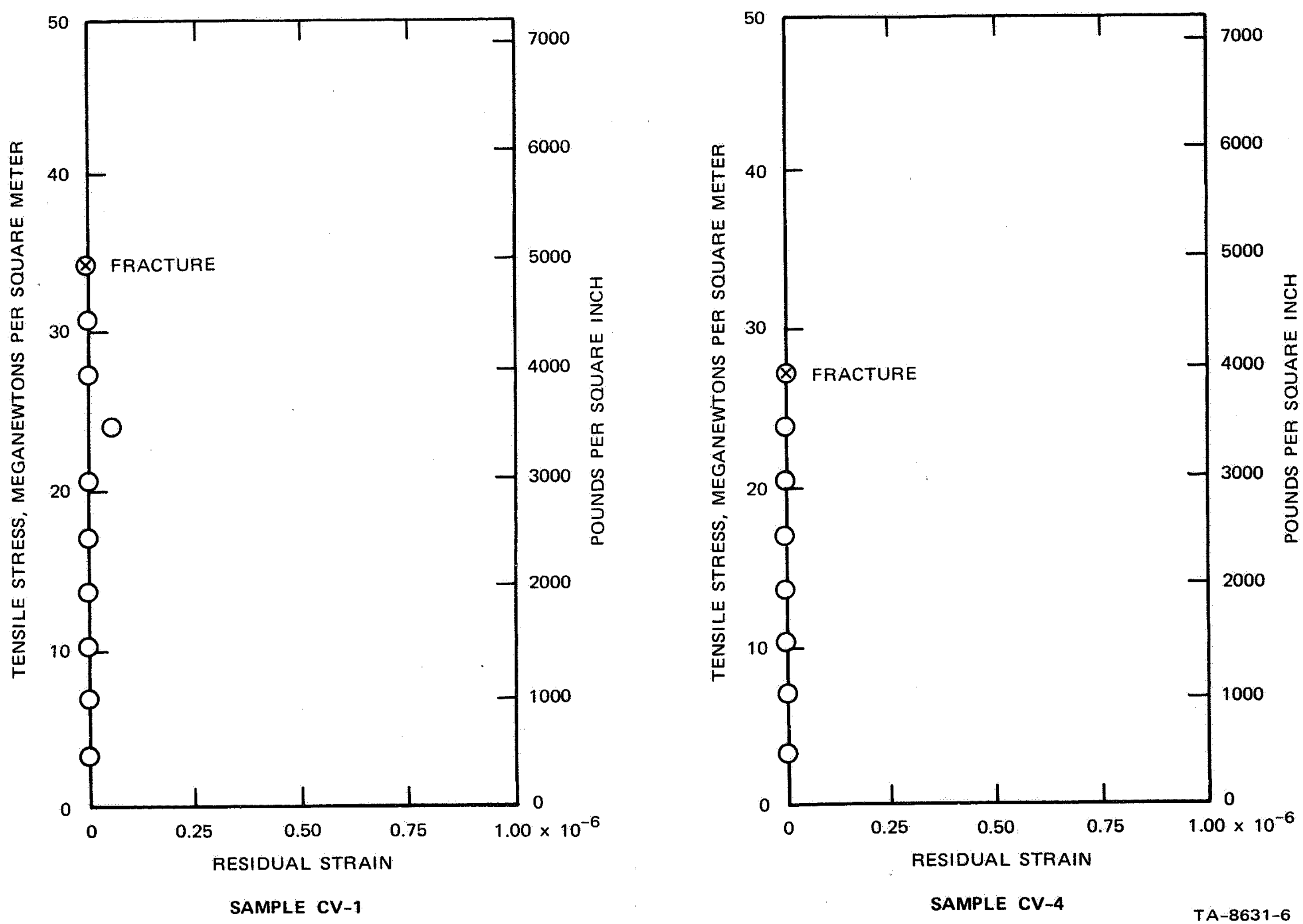

FIGURE 7 MICROSTRAIN MEASUREMENTS FOR TWO UNETCHED CER-VIT SPECIMENS

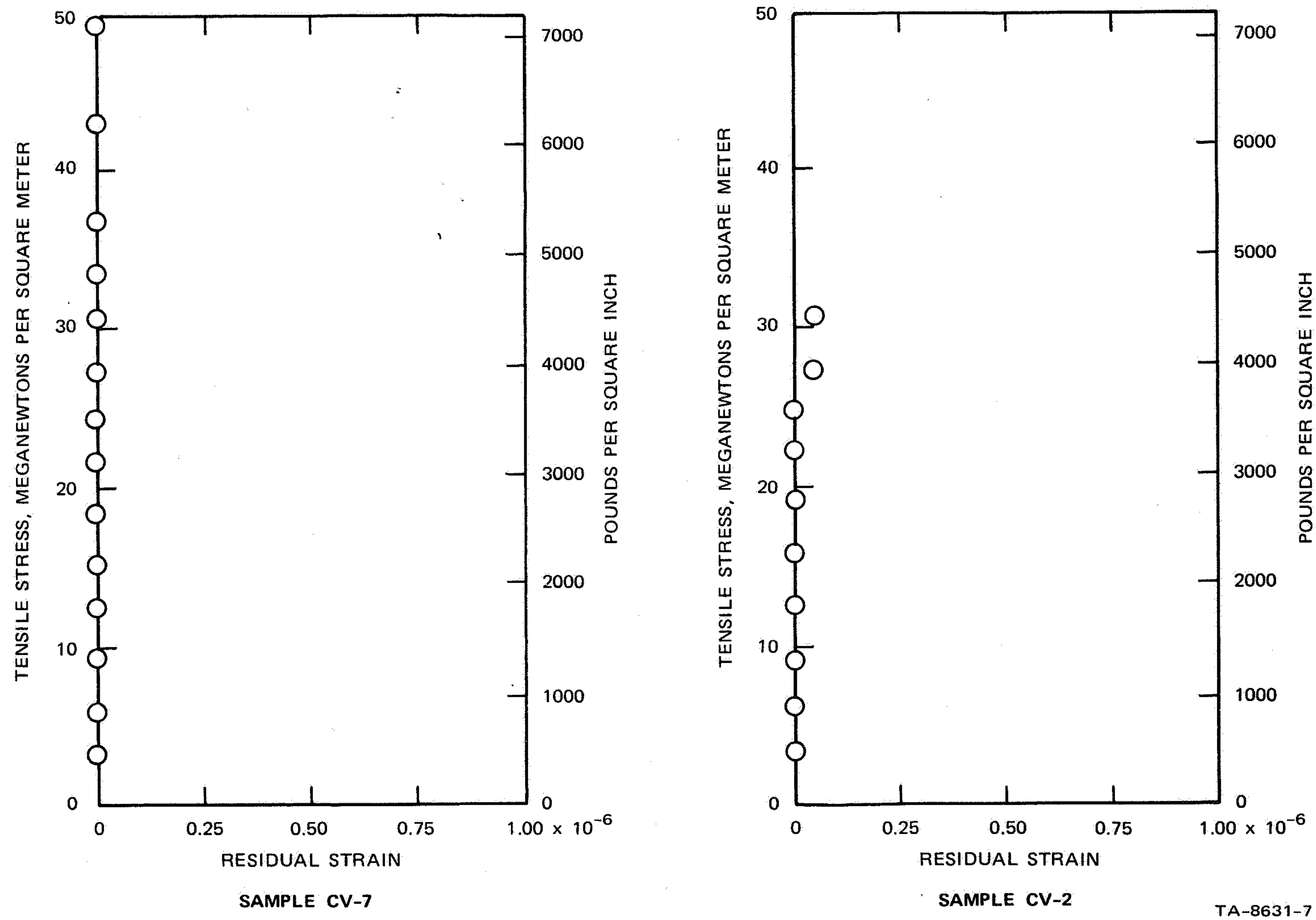

FIGURE 8 MICROSTRAIN MEASUREMENTS FOR TWO ETCHED CER-VIT SPECIMENS

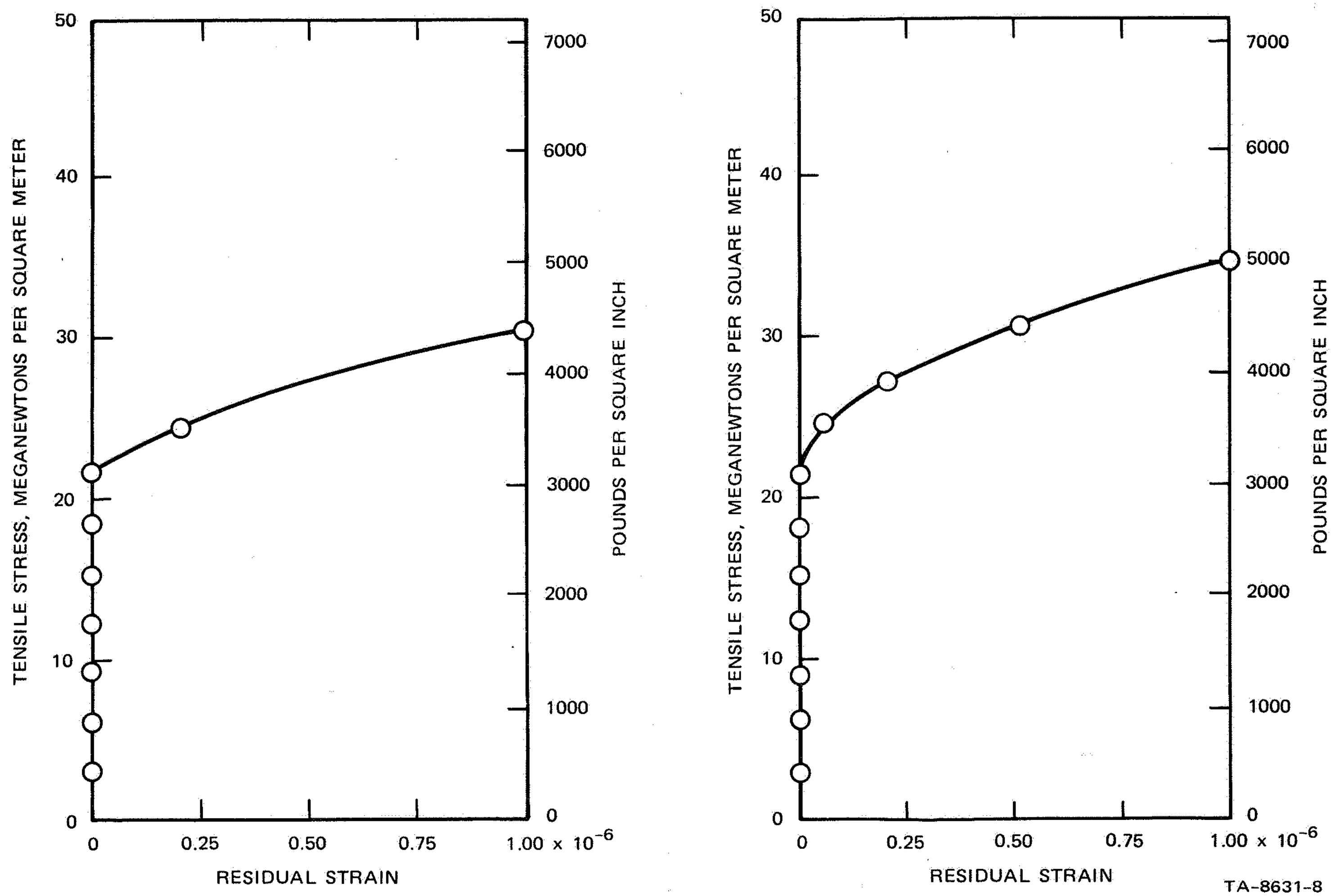

FIGURE 9 MICROSTRAIN MEASUREMENTS FOR TWO BERYLLIUM PLUS 5 WT % COPPER ALLOY SPECIMENS

DISCUSSION

No microyielding was detected for the three mirror materials--fused silica, ULE fused silica, and Cer-Vit. This is for fused silica and Cer-Vit in the etched and unetched surface conditions and ULE fused silica in an an etched surface condition. The test specimens were subjected to stress levels of 34 to 38 MN/m^2 (5000 to 5500 psi), and a strain sensitivity of 5×10^{-8} was used.

The only sample that exhibited any possible indication of microyielding was one of etched fused silica (FS-13); it showed a slight permanent deformation at stresses over 21 MN/m^2 (3000 psi). However, the results were particularly difficult to interpret, due to what appeared to be excessive anelastic recovery of the sample. The large anelastic recovery had the effect of reducing the strain sensitivity of the measurement to as low as 2×10^{-7}. This is because of the uncertainty of relating the strain of the 0.69 MN/m^2 (100 psi) reference level before and after loading. A strain of 2×10^{-7} is about the same magnitude as the strains recorded at the various stress levels when this fused silica specimen was tested. This is about the sensitivity limit of this one test. As such it is not possible to conclude that the results of this test indicate microyielding.

It is difficult to give an accurate account of the anelastic behavior. The ring test apparatus as presently used is a relatively "soft" test machine as compared with test machines using mechanical loading. Apparent anelastic behavior is introduced due to recovery of the rubber bag through which the load is transmitted. Also what may appear as anelastic strain is the equilibration of the water in the pressure lines. Although estimates can be made of the various effects by comparison of the behavior of samples of different materials, the precision of anelastic strain is low for this test technique.

Eul and Woods (Ref. 1) and Woods (Ref. 2) observed microyielding in unetched specimens, but not in etched samples. There were, however, two differences in the testing procedures and test conditions used in the previous studies:

(1) Strain sensitivity was five times greater

(2) Specimens were stressed to about twice the level reported here

Although tension, compression, and torsion (Ref. 1,2) testing all showed microyielding for the unetched specimens in the previous studies, the torsion tests were the most sensitive. If the microyielding is a surface controlled effect, it would be more pronounced in a torsion test, where the stress gradient is zero at the center of the specimen but increases to the maximum value at the surface. No such large stress gradients exist in tension tests.

Comparison of the present data does show discrepancies with the previous results. The earlier results from torsion tests can be summarized as in Table 1.

Table 1

MYCROYIELD RESULTS OF WOODS (Ref. 2) FOR UNETCHED FUSED SILICA: ULE FUSED SILICA: AND CER-VIT

Material	Approximate Stress to Produce 1×10^{-7} Residual Strain	Approximate Stress to Produce 5×10^{-7} Residual Strain
Fused silica	30 MN/m^2 (4350 psi)	50 MN/m^2 (7250 psi)
ULE fused silica	30 MN/m^2	50 MN/m^2
Cer-Vit	20 MN/m^2 (2900 psi)	40 MN/m^2 (5800 psi)

A strain of 10^{-7} is only twice the detection level used in the present study; however, a strain of 5×10^{-7} would certainly have been measured. Most of the tests in the present study involved loading the samples to 34 to 38 MN/m^2 (5000 to 5500 psi) which, according to the previous results, is not quite sufficient to produce a strain of 5×10^{-7}. However, one unetched fused silica sample (FS-11) was tested to 38 MN/m^2 (5500 psi) and exhibited no microyielding. One unetched Cer-Vit sample (CV-1), broke at about 34 MN/m^2 (5000 psi), again without microyielding.

The normal design stress limit for nonmetallic optical materials is 14 to 17 MN/m^2 (2000 to 2500 psi). This has been established not for microyielding, but rather as a safety factor to prevent fracture. These levels are about 50% the fracture stress that was observed for samples in this investigation. Using a stress limit of 17 MN/m^2 (2500 psi), no microyielding has been reported from any studies. The problem of microyielding is then important only if stresses greater than 17 MN/m^2 (2500 psi) are applied.

A mirror is better represented by the unetched surface condition. The results on the three nonmetallic mirror materials in the present study are in agreement with recent work by Goggin (Ref. 5). He used small mirror blanks 100 mm (4 in.) diameter for microstrain measurements on the same three materials. The mirror test specimens were polished by normal optical polishing techniques. This included surface grinding with various sized abrasives followed by final optical polishing. All processes were mechanical with no intermediate chemical etching treatments. The specimens had extremely smooth and flat surfaces, and were not etched before testing. The mirrors were then stressed by bending to apply a tensile stress to the polished surface. No microyielding was observed when surface stresses up to 69 MN/m^2 (10,000 psi) were applied. The strain sensitivity in these tests was about 10^{-8}.

Beryllium-Copper Alloy

The results observed for the alloy of beryllium plus 5 wt% copper is in close agreement with previous tests at SRI on other samples of the same composition. Permanent deformations were first measured at 24 to 28 MN/m^2 (3500 to 4000 psi), the specimen then showing substantial deformations at slightly higher stress. This trend is nearly identical to the results of Woods (Ref. 2) who tested a similarly fabricated alloy of beryllium plus 0.2 wt% iron. With the iron alloy the stress for onset of microyielding was about 10 MN/m^2 (1500 psi). The effect of the alloying additions is to raise the critical resolved shear stress for slip, thereby increasing the microyield stress of alloy.

CONCLUSIONS

1. Microstrain tensile tests with a strain sensitivity of 5×10^{-8} show that fused silica, ULE fused silica, and Cer-Vit are elastic when loaded to stress levels averaging about 34 MN/m^2 (5000 psi). This was observed both in samples whose surfaces were chemically etched to a depth of 0.10 mm (0.004 in.) and in unetched samples having mechanically ground surfaces.

2. These results agree with previous measurements (Ref. 1,2) which showed no microyielding in etched specimens, but do not agree with the results that showed microyielding in unetched samples.

3. Microstrain tensile tests (sensitivity 5×10^{-8}) on a well annealed, isotropic alloy of beryllium plus 5 wt% copper showed zero microstrain at stresses below 21 MN/m^2 (3000 psi) but showed a microstrain of 10^{-6} at 31 to 34 MN/m^2 (4500 to 5000 psi).

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ABSTRACT

Fused silica, ULE fused silica, and Cer-Vit are nonmetallic mirror materials being considered for use in diffraction-limited optical systems. A critical property of materials for these systems is a high microyield stress. Microstrain tensile tests were made to measure the microyielding behavior of the three materials and these are compared to an alloy of beryllium plus 5 wt% copper. The strain sensitivity for the tests was 5×10^{-8}.

It was found that fused silica, ULE fused silica, and Cer-Vit, having etched surfaces behaved in an elastic manner showing no microyielding. Tests on fused silica and Cer-Vit having unetched surfaces produced similar results, in that no microyielding was observed. The samples were stressed to 34 to 38 MN/m^2 (5000 to 5500 psi) or to their fracture point if it occurred at a lower stress. The beryllium-copper alloy showed a permanent strain after loading to above 21 MN/m^2 (3000 psi).

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