EFFECTS OF SURFACE POLISHING ON THE MICROSTRAIN BEHAVIOR OF TELESCOPE MIRROR MATERIALS

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SUMMARY

The principal objective of this program has been to determine the extent of removal by polishing techniques of the surface damage engendered by milling or grinding of candidate mirror substrate materials. This objective has been fulfilled with the presentation of quantitative figures of merit obtained when various surface treatments are employed.

Materials tested include fused silica (Corning 7940), titanium doped silica (Corning 7971), CER-VIT 101 (Owens-Illinois), and polycrystalline silicon (Exotic Materials).

Specimens were tested in torsional shear in four states of surface preparation: milled or coarse ground, etched, lapped or fine ground, and polished. Two schedules of lapping or fine grinding were investigated; "conventional" lapping in which material is removed only to the base of the pits produced by the previous abrasive, and "controlled" lapping in which the surface material is removed to a depth equal to three times the average diameter of the previous abrasive. Specimens subjected to the controlled lapping sequence exhibit significantly lower surface damage effects than those in the conventionally lapped condition. Final polishing of specimens after fine grinding reduces the surface yield still further, to within the uncertainty band presented by measuring and data reduction system precision limits.

INTRODUCTION

Studies of proposed orbital astronomical telescopes have indicated the need for diffraction limited optics in order to derive the maximum benefit from operating in the space environment. To insure diffraction limited performance of large space telescope mirrors, the strain stability of the substrate material must be optimized. Mirror substrates are initially shaped by milling or rough grinding operations. Previous microstrain tests on fused silica and devitrified glass in torsion (references 1 and 2) have

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ABSTRACT

Rough ground silicic mirror substrate materials have been found in previous investigations to exhibit significant surface yield. This effect was removed by surface etching, a procedure not normally employed in the finishing of optical telescope mirrors. The present work investigates effects of fine grinding and polishing techniques as well as graded etching. Torsional shear measurements of yield strain versus stress are made on four candidate mirror substrate materials: polycrystalline silicon, ULE silica 7971, CER-VIT 101, and fused silica 7940. Commonly employed fine grinding and polishing practices are shown to remove a major portion of the surface yield found in rough ground mirror substrate materials.

indicated that surface damage from rough grinding imparts a permanent yield characteristic to the surface. Removal of a few tenths of a millimeter of surface by acid etch removes the yield characteristic, leaving the base material without appreciable permanent deformation at all loads up to fracture.

The portions of a telescope mirror not actively employed for optical reflection are often acid etched to remove surface damage. The resulting surface roughness is unsuitable for the active reflecting surface, which is prepared by mechanical lapping with fine grinding and polishing abrasives. The surface yield characteristic produced by this treatment had not been determined, and questions have been raised as to the suitability of presently employed fine grinding and polishing procedures.

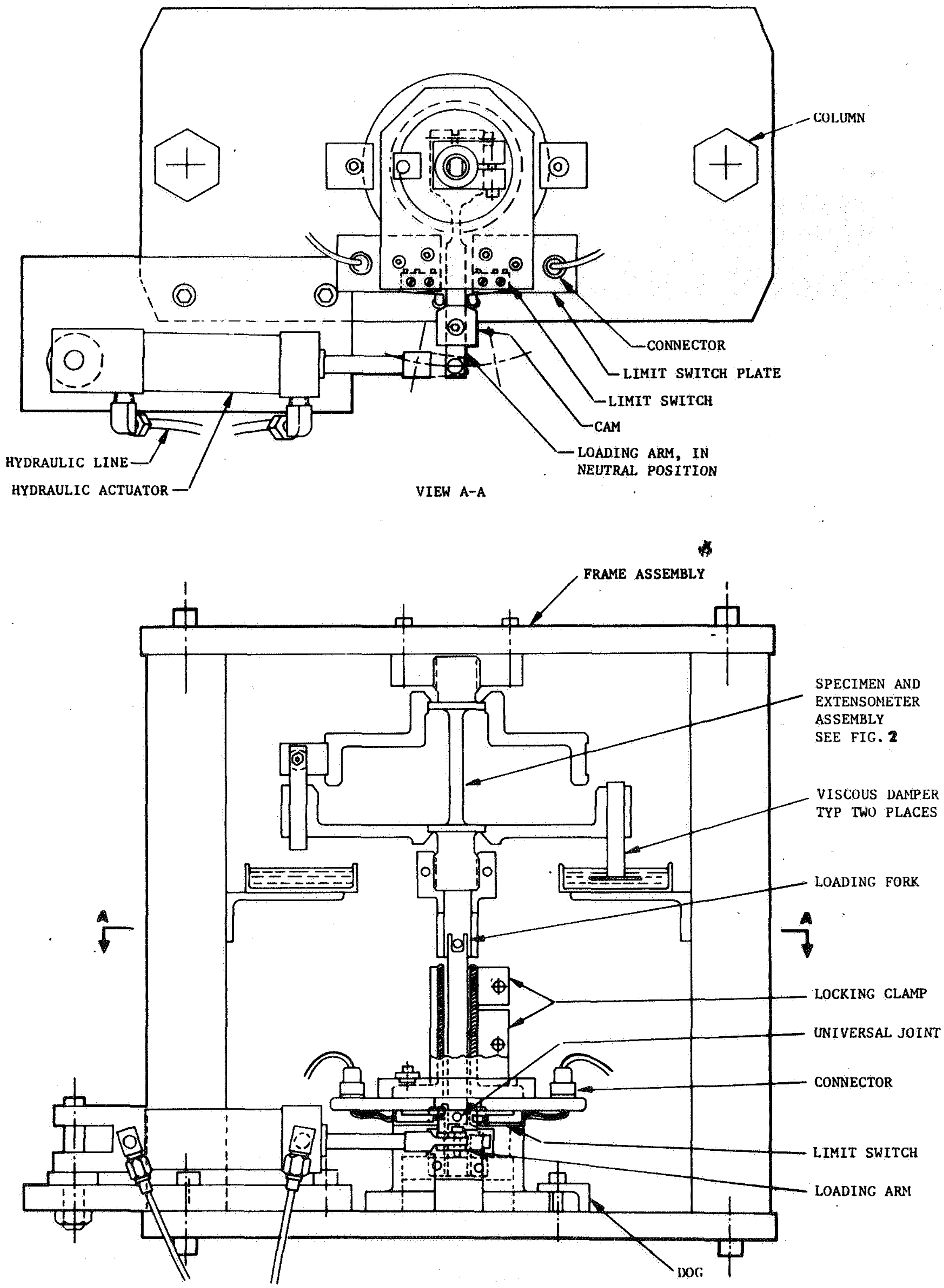
The first objective of the present program was to determine the depth of surface damage from coarse grinding. By testing specimens after successive etches of the order of 0.01 mm removal each, the gradual improvement of characteristics from the rough grind to the fully etched base material was expected to be revealed, and the effective depth of damage determined.

The second, and major objective of this program was to determine the extent of surface damage removable by graded lapping or fine grinding techniques and the establishment of a fine grinding and polishing schedule or recipe to achieve an optimum surface. Tests of "conventionally" lapped specimens, where material is removed only to the base of previous pits, are compared with specimens lapped to greater depths.

The third objective is the determination of microstrain behavior of polycrystalline silicon mirror substrate material, in the ground, etched, and polished conditions.

EXPERIMENTAL APPARATUS

The experimental apparatus used in this program is the torsion test equipment described in reference 2, with minor circuitry modifications to simplify operation. The basic loading and readout systems are unchanged. A manually operated hydraulic pump operates a hydraulic cylinder which loads the specimen through a strain-gaged lever arm. The strain gages are monitored to determine load level. Load release is effected by valving a pressurized accumulator under the automatic control of deflection limit switches. When load is released, the designed-in backlash of the loading system completely uncouples the loading train from the specimen and the specimen is free to relax without friction or load train influence. A sketch of the torsion microstrain apparatus is shown in figure 1. A concentric double walled thermal enclosure with two-zone thermal control system maintains the testing apparatus at a near-constant temperature of 306K.



NOTE: FOR CLARITY SOME PARTS OF THE ASSEMBLY ARE SHOWN OUT OF THEIR TRUE LOCATION.

Figure 1: TORSION MICROSTRAIN APPARATUS

The extensometer, in the form of instrumented lava cups, is cemented to low stress shoulders on the specimen between the end loading sections and the test section (see figure 2). Linear variable differential transformers, mounted around the periphery of the cups, are summed to read out differential angular motion and to discriminate against differential linear or bending motions. Provisions are made to monitor bending motions of the specimen.

Readout of specimen loading and deflection is facilitated by a digital voltmeter and printer. Upon release of load, an electronic programmer triggers the voltmeter-printer to log recovery data at decimal intervals of 5, 50, 500, and 5000 seconds until reset for the succeeding load. This quasi-logarithmic time scale is chosen to facilitate data reduction. The sequencer resets automatically at the end of a present interval, and activates an alarm to alert the operator to initiate the succeeding loading sequence.

TEST SPECIMENS

Test specimens, as shown in figure 3, have a conventional cylindrical test section terminated by low-stress shoulders to which the extensometer is cemented. Threaded, large cross-section ends allow secure fastening to cemented-on load grips.

Materials chosen for the test were as follows:

Material	No. of Blanks	Specimen Numbers
Polycrystalline Silicon Mirror Blank Grade	4	201-204
7971 ULE Fused Silica Mirror Blank Quality	6	211-216
CER-VIT C-101 Premium Grade Mirror Blank Quality	6	221-226
7940 Fused Silica Mirror Blank Quality	6	231-236

Specimen Preparation

Test specimens were machined from each material upon receipt from the supplier with a 120-grit diamond wheel. Prior to testing, specimens were subjected to a heat treatment of 810 K for one hour with oven cooling. Although this treatment cannot be considered an annealing treatment for the base material, it has been found adequate to stabilize surface stresses. An attempt was made to provide an annealing treatment in vacuum to the first two silicon specimens, but in the process a eutectic was formed with the

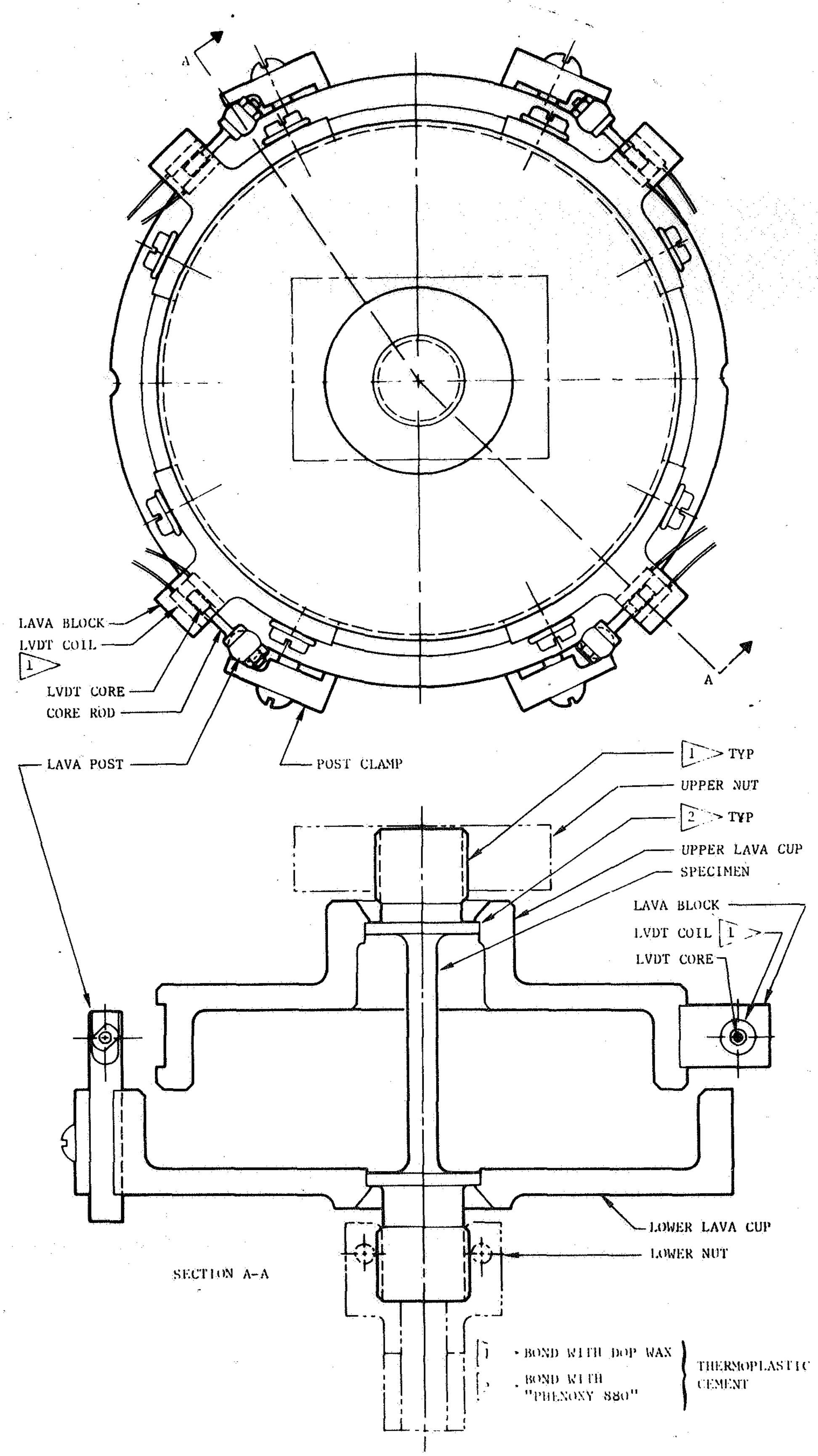
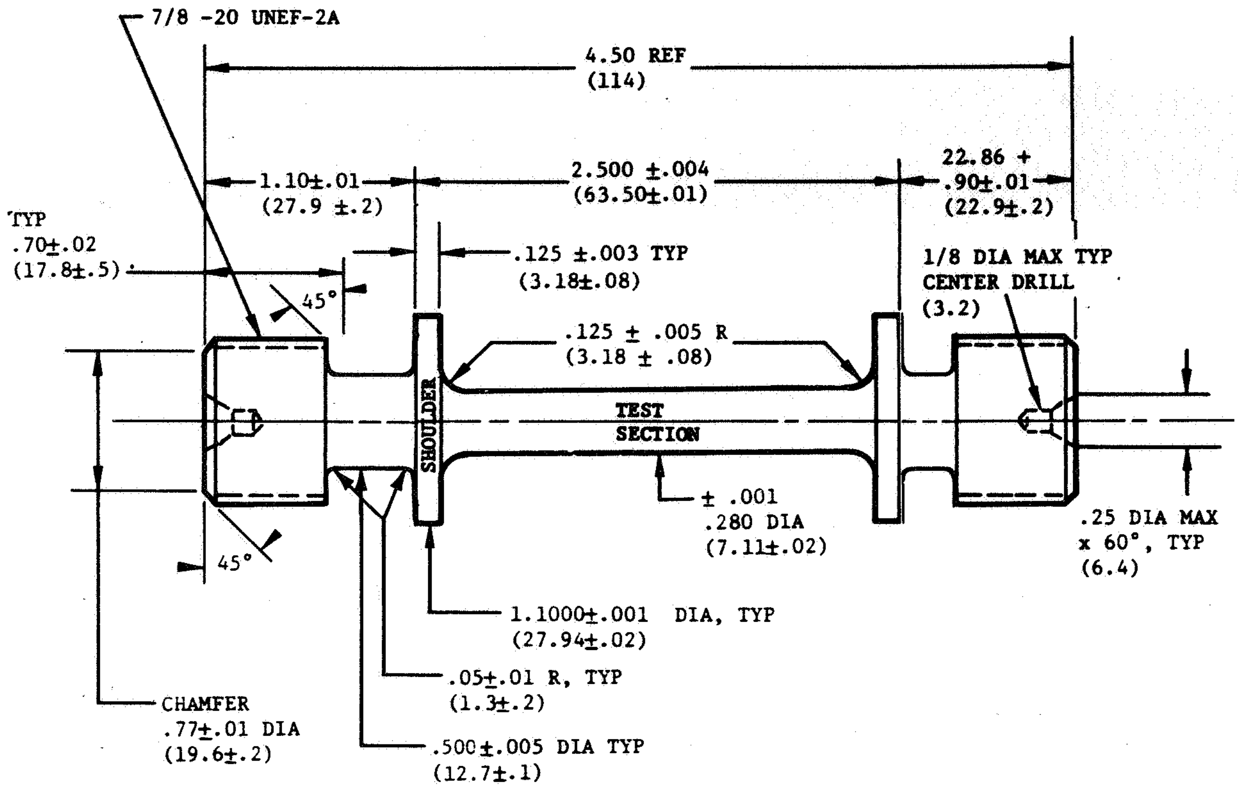


Figure 2: SPECIMEN - EXTENSOMETER ASSEMBLY



ALL DIMENSIONS IN INCHES (mm)

PART TO BE STRESS RELIEVED BEFORE AND AFTER MACHINING.

BREAK ALL CORNERS .005 to .010 R (.1 to .2)

ALL DIAMETERS TO BE CONCENTRIC, WITH END CENTERS WITHIN .001 TIR. (.02)

Figure 3: TEST SPECIMEN SKETCH

steel holding fixture and the specimens were destroyed. Thereafter only the surface stabilization heat treatment was employed.

Surface Treatment

After an initial test in the rough ground condition, specimens were subjected to an acid etch. Etch formulations peculiar to each material are listed in Appendix A. Tests of specimens etched to moderate or very shallow depths showed a drastic improvement over the rough ground specimens. Subsequent etching produced very little change, and no further attempt was made at gradation of etch.

The basic intent of this work was to evaluate conventional and current practices of mirror surface preparation. Two fine grind procedures were chosen for evaluation, adapted from the work of reference 3 wherein surface tensile strength of plane mirrors was related to removal of surface microcracks. The "conventional" fine grind procedure employed in the past employed each abrasive only to the point of removal of pits from the previous abrasive. The above reference calls out a schedule of surface removal for each abrasive corresponding to 60% of the average diameter of the previous abrasive. The controlled fine grind schedule on the other hand, calls for removal of three times the diameter of the previous abrasive. In this program, the choice of abrasive material type was unimportant to the test results, and was largely determined by shop personnel preference for optimum cutting rates. Grinding and polishing schedules followed in this program are listed in Appendix A.

Preparation, testing and surface treatment histories of each specimen are listed in Appendix B.

TEST PROGRAM

At the start of this program, a series of 32 tests was scheduled to determine the torsion microyield properties of silicon, fused silica, and devitrified glass mirror substrates. Surface conditions to be investigated included coarse ground (milled), etched, fine ground (lapped), and polished. This schedule has been followed as closely as possible, although problems with specimen breakage and fabrication procedures have caused a few deviations. Appendix B lists the histories of test specimens.

Test Procedures

The specimen under test is cemented to the extensometer cups with a thermoplastic cement, Phenoxy 880, which has a working temperature of 440 K. Threaded loading grips are attached to the threaded ends of the specimen with a silicate-loaded wax which has a working temperature of 370 K. With this combination, secure, strain-free attachment is made in successive operations without mutual interference.

After installation in the torsion test apparatus, the specimen and extensometer are given a minimum of 24 hours to come to thermal equilibrium. Temperature in the inner test chamber is controlled to 306K with a stability of $\pm$.005 K.

Specimen loading is accomplished in logarithmic increments approximating the fifth root of ten, from 0.7 to 30 meganewtons per square meter. Successive increments are made in alternate directions to maintain a check on the system zero reference. Load is applied by operation of a hydraulic cylinder with a hand pump. When the desired load is reached, the load and extensometer readings are recorded. The load is released over a period of two seconds or less by valving a pressurized accumulator to return the hydraulic cylinder to zero position, where a limit switch stops the return motion automatically. Digital voltmeter recordings of the extensometer output are programmed by an electronic timer at prescribed intervals to yield a quasi-logarithmic time sequence of readings.

Resistor standards within the equipment are used as references for both loading torque and angular deflection values. These standards are calibrated before the start of the test program by comparing output signals from resistor stimulation with signals generated by dead weight loading (torque) and optical autocollimator-measured angular deflections. The resistor standards are used to stimulate torque and angle sensors and standardize gains of the measuring equipment at the start of each test run. Digital recordings of these stimulations are employed by the data reduction computer program as calibration factors.

Yield measurements in the range of one microstrain and below are accomplished by loading the specimen, releasing the load, and measuring the resulting offset. In this range significant time-dependent or viscoelastic strain may complicate the determination of permanent yield. Some materials, such as the silicas, recover rapidly and may be measured without extrapolation within ten to fifteen minutes after each load release. Others such as CER-VIT have extended decay periods, and must be extrapolated to final end point if reasonable test times are to be employed. Computer programs described in the next subsection, "Data Reduction," are employed to accomplish the extrapolation.

Data Reduction

Detailed reduction of the printed-out test data is accomplished with the computer program of Appendix B of reference 2. Minor changes have been made to accommodate changes in computer software since the previous test program. Zero and gain levels of the test equipment, established at the start of every run, are applied to the reduction of the test data. Viscoelastic relaxation profiles of each specimen are extrapolated to determine permanent microyield end points. Plots of viscoelastic decay are made on the computer printout for convenient visual examination.

A review of the data derived from the first test runs by the above computer program indicated that minor thermal drifts and random noise in the recorded test data were adversely affecting the extrapolation of viscoelastic decay curves to their end points. At the same time, it was noted that the most consistent derived data was obtained when the viscoelastic decay could be closely represented by an exact time reciprocal function. An expression involving intervals of time after release of stress was derived (see Appendix C) and applied to previously recorded test data. A significant improvement in derived data consistency and reproducibility was obtained. Accordingly, a revised and shortened data reduction computer program (Appendix D) was coded for processing all the specimen runs for microyield data.

Once the individual loading end points are determined, they are plotted vs. time of day on a linear scale, as in figure 4. A series of ten to fifteen loadings customarily takes from two to twelve hours to complete, depending upon the material, its surface condition, and the desired end point accuracy. Over such time intervals, slight zero drifts of the order of one to ten nano-strain are difficult to avoid. With the stress loads and the yield values alternated at each succeeding load, zero offset and zero drift are readily evaluated from the time plot and subtracted from the end point data. Although this should furnish true yield data, another systematic factor enters the picture when specimens of very low yield are tested. Brittle specimens which have had 0.2 mm or more etched or lapped from their test section diameter, exhibit a "negative hysteresis" or "negative yield" effect proportional to the first power of applied torque. This effect is repeatable but varies with material. The "moduli of negative yield," normalized for specimens of 7.6 mm diameter are as follows:

Material	Modulus, TN/m^2
Silicon	- 24,000
7971 ULE Silica	- 6,200
CER-VIT	- 5,700

It is illogical to assume that this effect occurs in the test section of the specimen, "over-reacting" to the applied stress. The deflections, roughly one millionth of the deflection under load, are most probably the result of small elastic and viscoelastic strains in the specimen shoulder, reacting with the cemented attachment to the extensometer cups. As this attachment does not change with the test section diameter, the effect is more properly described as an angle vs. torque relationship rather than strain vs. stress. To determine the magnitude of the effect in terms of apparent negative strain vs. stress in a given test, the above moduli are multiplied by the fourth power of the ratio of the reference diameter (7.6 mm) to the test section diameter. (See Appendix E.) Test stress levels are divided by the resulting applicable modulus to yield the instrumental "negative yield" strain. These values of strain are added to the extrapolated zero-corrected yield strain values and the result is the test section true yield strain (negative yield effect eliminated). As the 7940 silica was not tested in the etched condition, a negative yield modulus was not determined, and the modulus for ULE silica was used to reduce the final polish data.

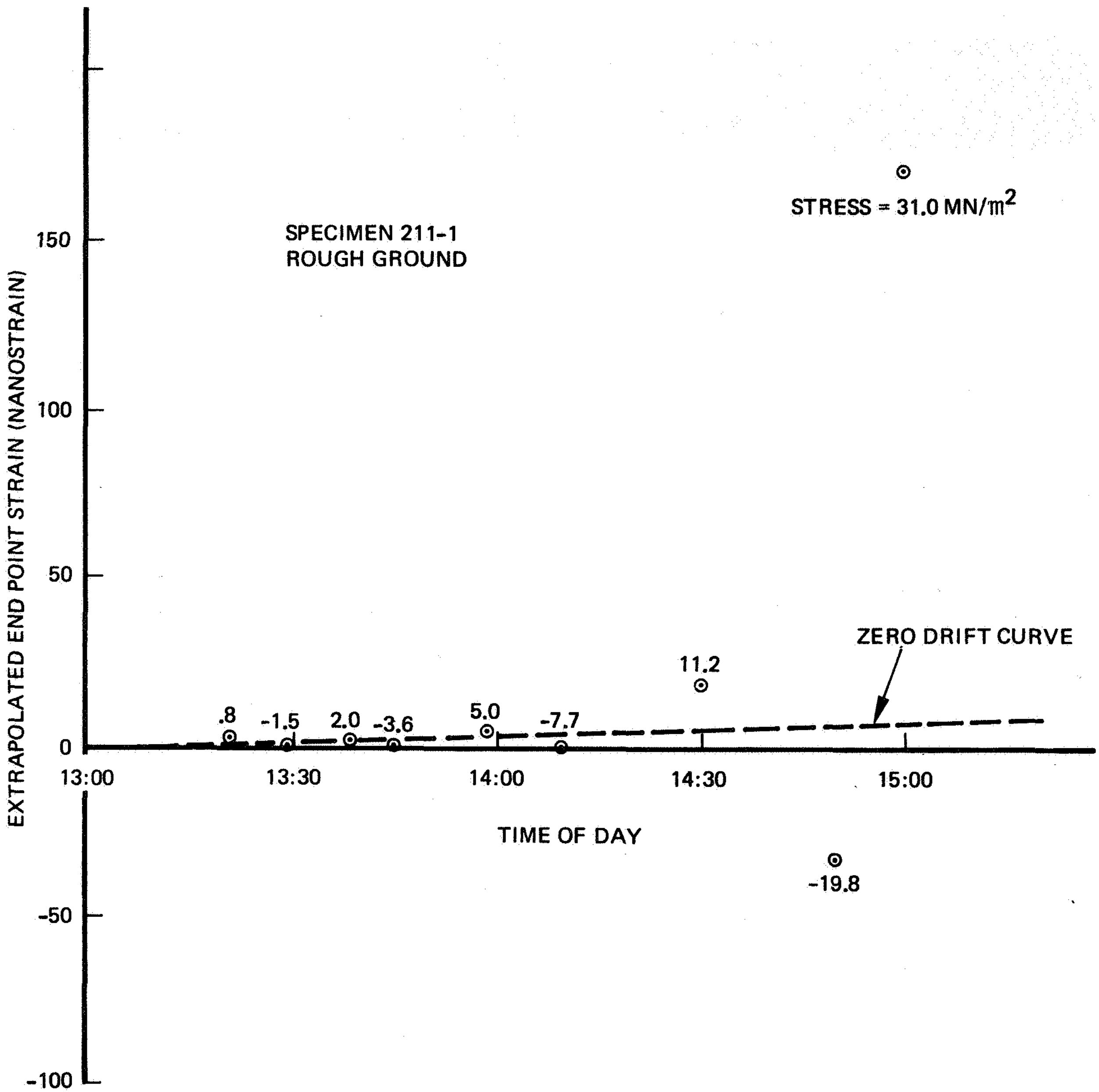


Figure 4: ***EXTRAPOLATED END POINT PLOT***

These corrected end point yield strain values are plotted vs. stress on a log-log plot. A representative plot is shown in figure 5. The curves so formed tend to be straight lines with a slope of one-half for all materials tested in this program, suggesting that the yield varies as the square of the stress. As this appears to be a general rule for the brittle materials of this test program, a pseudo-modulus of yield M_y such that $M_y = 10^{-4} \cdot$(stress @ 10^{-8} yield) was developed to allow convenient comparison of materials behavior. That this is not a true base material modulus is apparent from its complete dependence upon surface condition and specimen geometry. This modulus is finally corrected for test section diameter as described in Appendix F.

The magnitudes of delayed elastic strain of the materials tested differ widely in magnitude, but exhibit a strong proportionality to stress divided by time after release of stress. A modulus of delayed elastic strain M_{DE} such that

$$M_{DE} = \frac{\text{Stress}}{100 \quad (\varepsilon_{50} - \varepsilon_{100})}$$

where

$$\varepsilon_{50} = \text{strain at 50 seconds after release of stress}$$

and

$$\varepsilon_{100} = \text{strain at 100 seconds after release of stress}$$

was utilized to compare viscoelastic behavior of different materials with various surface conditions.

A third modulus, the familiar elastic shear modulus G, was derived from specimen deflection under load for the materials tested. This adds little to basic knowledge of the materials, but provides a comparison to the other moduli.

RESULTS

In the following description of results, stress is expressed in metric units of newtons per square meter. A conversion factor to the English system is:

1 meganewton per square meter = 145 psi

Two candidate mirror substrate materials, CER-VIT and 7971 silica, were tested in various states of surface conditioning, including rough milling, etching, and fine grinding. Silicon was given cursory tests in milled,

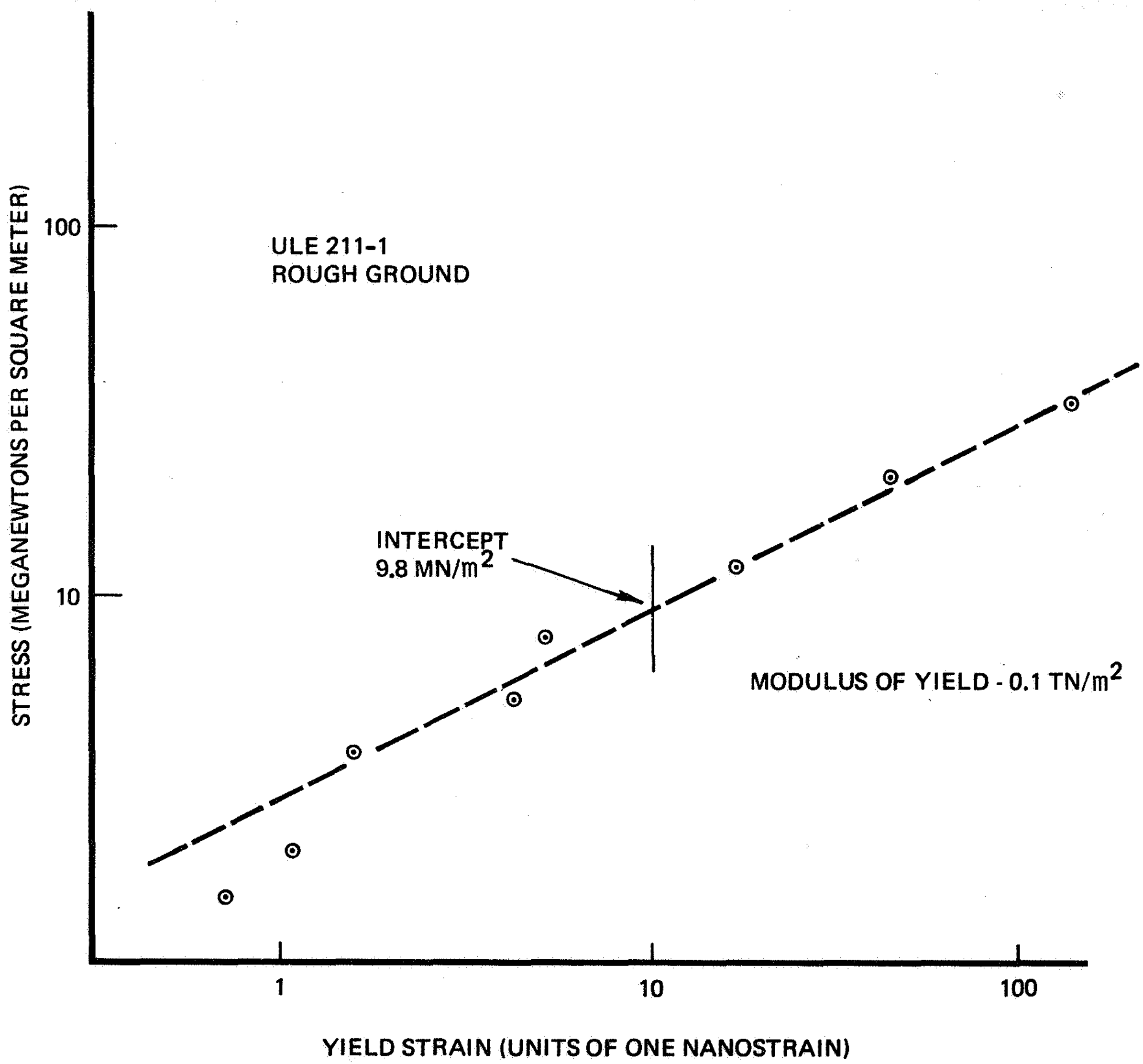


Figure 5: YIELD STRAIN VS STRESS

etched, and partially fine ground conditions. All four candidate materials were tested after fine grinding and polishing. Results are shown in bar charts in which the height of each box represents the spread of the data.

Microyield

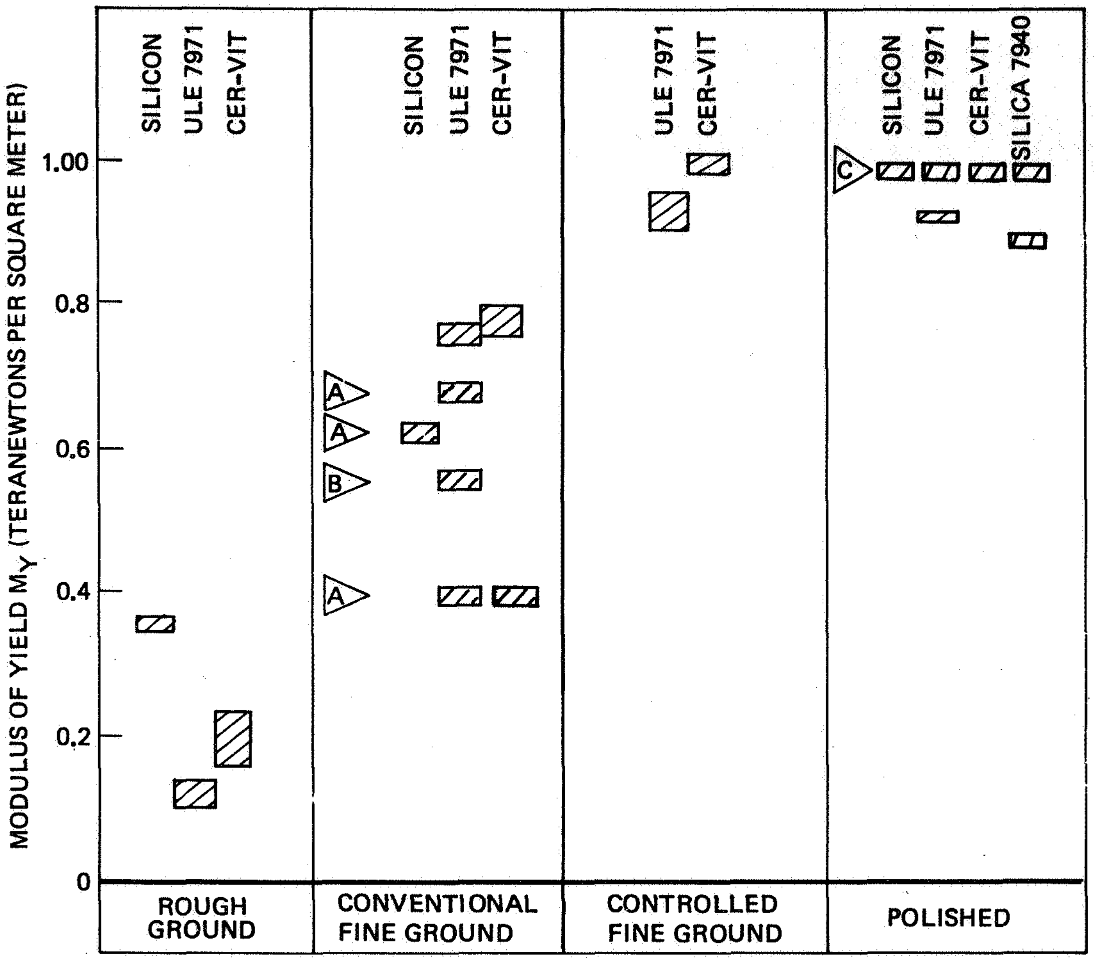
Initial tests of rough milled specimens reproduced the relatively large values of yield obtained in earlier programs for this surface treatment (reference 1). None of the specimens were taken to the 1 x 10^{-6} yield strain point, however, as this is generally beyond fracture, and the specimens were intended for retesting after each of several surface treatments. Surface etching of the specimens produced a dramatic improvement, even with relatively small amounts (0.004 mm) of material removal. Etched specimens appeared to have virtually complete recovery, revealing the "negative hysteresis" characteristic of the testing apparatus. This response characteristic, assumed to correspond to that of a perfect specimen, was then utilized as a zero yield reference in all ensuing data reduction.

As tests proceeded, some of the milled or rough ground specimens produced results inconsistent with other tests. An intensive review of shop practices revealed that the machinist had superficially lapped the specimens to improve surface finish. An occasional specimen would receive more lapping than others. Regrinding these specimens with a diamond wheel re-established the basic rough ground yield levels.

The "conventional" fine grind produces a marked improvement of yield characteristic over that of the rough ground specimens. The yield, however, is still measurable. Controlled fine grinding techniques reduce the yield still further, approaching the limit of precision of this testing equipment and data reduction process. After a subsequent polish, the yield characteristic approaches that of etched specimens, which for the purposes of this application, represents perfect recovery.

Results of yield measurements in terms of yield modulus are shown on the bar chart of figure 6. Yield modulus, rather than yield vs. stress, is presented to better display relative differences of materials and surface treatment. For significance of this parameter with respect to large diffraction limited mirrors see "Yield" in the section titled "Discussion." This modulus of yield is based upon a square law response to surface stress, and has exact numerical meaning only to torsional shear of 7.6 mm diameter specimens. The development of an exact analytical formulation relating these results to the performance of large mirror substrates is not a part of this program, and has not been attempted.

Silicon is seen to have superior yield characteristic in both the rough ground and polished states. The one fine ground specimen tested was not scheduled, and no information was available as to the amount of surface removal in the fine grind. The results from this fine ground specimen therefore cannot be compared directly with other materials.



YIELD STRAIN = $(\text{STRESS}/M_Y)^2$

NOTES:

A — FINE GROUND WITH 600 MESH BORON CARBIDE UNSCHEDULED

B — FINE GROUND WITH 600 MESH BORON CARBIDE TO A DEPTH OF 0.04 mm

C — VALUES GREATER THAN 1.0 ARE SHOWN AS 1.0

Figure 6: MODULUS OF YIELD

CER-VIT has a larger value of yield modulus (lower yield) than the ULE silica with similar surface treatment, but the difference is relatively small. A few tests of ULE silica in a partially fine ground condition, fabricated in most cases without measurement of surface removal, imply gradual improvement of yield characteristic with depth of fine grinding.

After rough grinding, partial fine grinding, or conventional fine grinding the heat treatment discussed under "Specimen Preparation" in the section titled "Test Specimens" was required to stabilize surface stresses and to provide reproducible test results. After controlled fine grind or polish treatment, however, adequate stability was obtained without heat treatment (heat treat only stabilizes surface cracks).

Delayed Elastic Strain

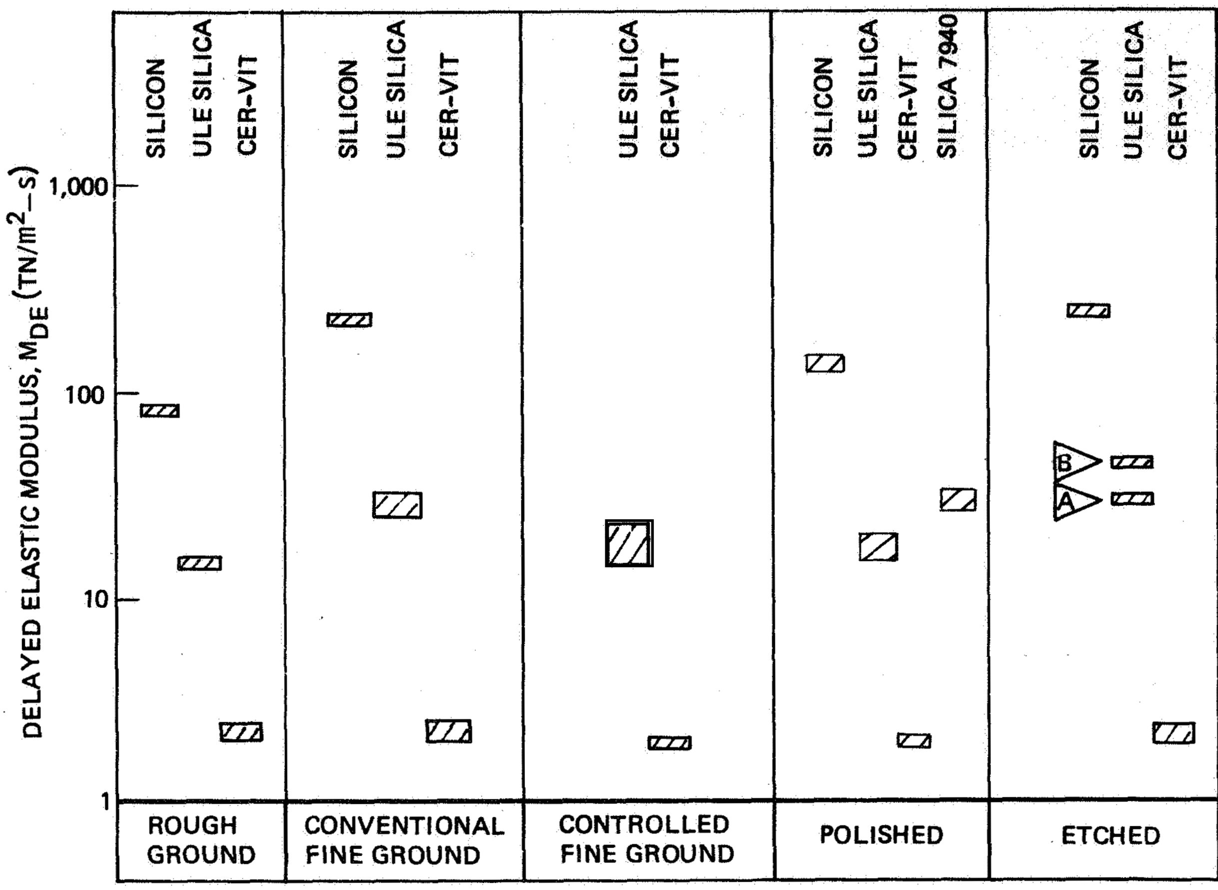
The sensitivity and resolution required of the test equipment to measure the small yield strains of this test program strongly illuminated the delayed elastic strain characteristics of the test materials. Delayed elastic, visco-elastic, or time dependent strain, as it is variously termed, is obviously a true body material characteristic as opposed to surface effects. It varies by orders of magnitude between materials, and is only moderately influenced by surface treatment. Figure 7 shows in bar chart form the range of values of delayed elastic modulus measured for the test materials of this program.

Silicon is seen to have the highest modulus (fastest return) and CER-VIT the lowest modulus of delayed elastic strain (slowest return to rest position). The silicas take a position close to the geometric mean between these two responses.

Delayed elastic strain in CER-VIT is virtually independent of surface treatment, probably because the intrinsic low modulus overpowers any surface effect. As the modulus of the material increases the surface effects are more easily observed, as in the silicas and silicon. The rough ground surfaces appear to lower the effective modulus as compared to the etched surfaces. Differences of modulus with depth of etch is apparent in ULE silica. The apparent lowering of the delayed elastic modulus with controlled fine grind and polishing on the silicas and silicon may be due to effects of the viscous damping system attached to the extensometer becoming appreciable at short time intervals after release with reduced-diameter, low-stiffness test sections. It would appear unlikely that the fine grinding or polishing treatments themselves are responsible.

Elastic Shear

Values of elastic shear modulus (G) were derived from the specimen deflections under load for the four test materials. Within the reproducibility of the test equipment, this modulus was independent of specimen treatment or surface condition. The modulus is supplied here to compare



NOTES:

A ETCHED 5 μm

B ETCHED 30 μm

$$\text{STRAIN} = \frac{\text{STRESS}}{M_{DE}\ t}$$

t = TIME AFTER RELEASE OF STRESS

Figure 7: DELAYED ELASTIC MODULUS

with the other moduli measured. Figure 8 shows the range of values determined for this modulus of each material.

DISCUSSION

Yield Strain

The values of modulus of negative yield on all materials tested in this program exceeded 0.8 teranewton per square meter for specimens subjected to controlled fine grind and polishing treatment. The first obvious question to be asked is, what does this mean in terms of a large mirror substrate. Because the effect is apparently caused by surface defects, and will be influenced strongly by substrate geometry, exact values will require extensive analysis and computation. It should be valid to assume, however, that the test specimen geometry utilized in this program is much more sensitive to the surface yield effect than any practical large mirror substrate. For a given stress level, therefore, the apparent yield strain exhibited by this test specimen geometry should be an upper limit to the apparent yield strain of a large mirror substrate. If we assume a maximum peak shear stress level in the mirror surface of 80 meganewtons per square meter (11,600 psi), which approximates the breaking strength of most such brittle structures, our specimen will have a yield of less than 1×10^{-8}. A structure of maximum dimension of 3 meters and a uniform strain of 1×10^{-8} will exhibit a deflection of 30 nanometers, or roughly 1/130 the wavelength of 400 nanometer visible light. This dimension is less than common criteria for figuring a high grade diffraction limited mirror.

As stated above, this effective yield is regarded as an upper limit and a practical mirror substrate should exhibit less yield by more than an order of magnitude. As all four materials tested had yield moduli exceeding 0.8 TN/m^2 when the surface is carefully fine ground and polished, the surface yield criterion should not be important in the choice of a substrate material.

Delayed Elastic Strain

Delayed elastic strain is important in an optical system when accurate figure is required a short time interval after being subjected to high stress levels from mechanical or thermal loading. Let us consider CER-VIT, which has the lowest delayed strain modulus (two teranewtons per square meter-second) of the four materials tested. When subjected to a stress of 80 meganewtons per square meter, which approaches its ultimate strength in shear, it will retain a strain of 4×10^{-5} one second after load release, 6.7×10^{-7} at one minute, and 1.1×10^{-8} at one hour. Assuming worst case, as in the previous section, a three meter CER-VIT mirror substrate subjected to breaking strength load should recover to within the diffraction limit criterion within one hour. For a silica mirror (modulus = 40 TN/m^2 - sec) the recovery would take place within three minutes, and for silicon

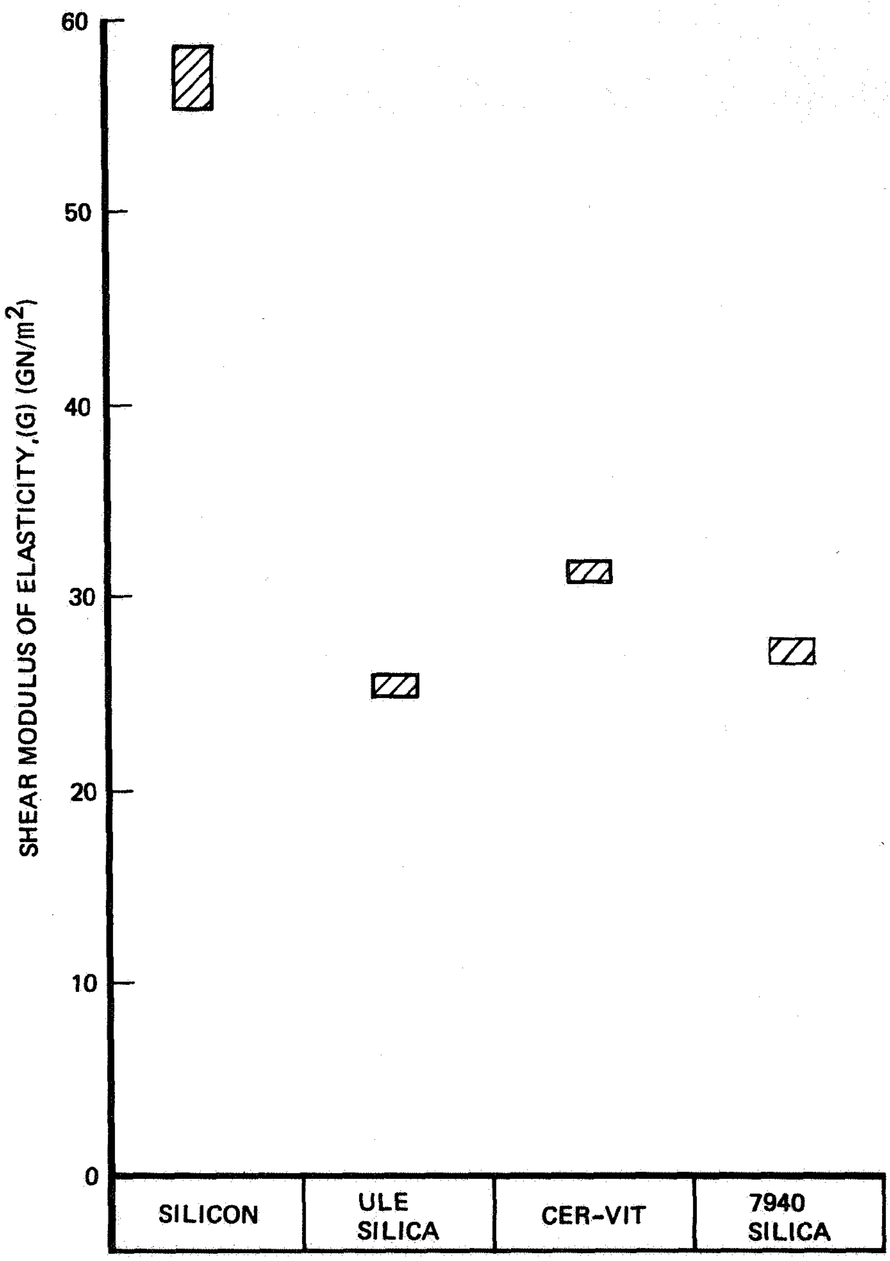


Figure 8: ***ELASTIC SHEAR MODULUS***

(modulus = 200 TN/m^2 -sec) less than one minute. Depending upon the intended usage of the mirror, the delayed elastic modulus may or may not be a criterion for selection. It should be noted at this point that viscoelastic properties are often temperature dependent. The measurements of this program were all taken at a temperature of 306 K. If a temperature appreciably lower than this is contemplated, and if rapid recovery from mechanical loading is important, the delayed elastic effect of substrates of interest at lower temperature should be investigated.

Elastic Strain

The modulus of elasticity is not a new concept, and is adequately well known for the materials of interest making the data on this parameter presented in this report relatively unimportant. It does, however, emphasize the superiority of silicon in resisting deformation from mechanical stresses. However, the current processes of production of silicon substrates limit available mirror size, and may rule out a large monolithic silicon structure. The differences in elastic modulus between silica and CER-VIT are not large, and are not a major factor in the choice between these two substrate materials.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

(1) The primary objective of this program, which was to determine the effects of fine grinding and polishing on the microstrain behavior of mirror substrate materials, has been accomplished. Effects of two fine-grinding schedules have been investigated and the results compared quantitatively. The controlled fine grinding sequence of Reference 3 has been shown to be superior for minimizing surface microyield.

(2) When the controlled fine grinding sequence of Reference 3 is employed, the predicted maximum yield strain of mirror substrate materials investigated is small with respect to criteria for diffraction limited mirrors.

(3) Where the substrate material is not subjected to appreciable loading shortly before its intended use, the delayed elastic strain behavior is unimportant at normal laboratory ambient temperature or above. The three classes of materials tested in this program, CER-VIT, vitreous silica, and silicon, differ widely in this characteristic.

(4) Of the materials tested in this program, silicon is outstanding in its elastic, delayed elastic, and surface yield properties.

(5) Of the materials other than silicon tested in this program, no clear superiority is evident. CER-VIT shows drastically greater delayed elastic strain effects, but this is of little significance in conventional telescope mirror usage.

Recommendations

The objectives of this program have been met without significant unanswered questions. This program was limited in its objectives, however, and several areas remain to be explored. These are detailed as follows:

(1) Thermal effects. Delayed elastic strain, although relatively unimportant at 300 K, may be significant at lower temperatures. Determination of the thermal dependence of this characteristic, especially for CER-VIT, is recommended.

(2) Creep. Past evaluation of the viscosity of glassy materials in the 300 K temperature range is at best controversial. Extension of currently accepted elevated temperature viscosity figures to temperatures approaching 300 K is recommended.

(3) Beryllium. Originally intended for test in this program, copper alloys of beryllium were removed from consideration by limitations of funding and calendar test time. Beryllium and its alloys are still of interest for mirror substrate fabrication. The precise nanostrain behavior of these materials remains to be documented, and should be considered.

APPENDIX A

SPECIMEN SURFACE TREATMENT

Etch Formulations

Reagent \ Material	Silicon	ULE Silica	CER-VIT
HF	14%	50%	10%
HNO_3	32%		
H_2SO_4			10%
C_2H_3OOH	27%		
H_2O	27%	50%	80%

Fine Grind Schedules

Grit	Grit Diameter Micrometers	Surface Removal Micrometers
Conventional Fine Grind		
#400 Silicon Carbide	45	60
#225 Aluminum Oxide	22	28
#125 Aluminum Oxide	12	18
#95 Aluminum Oxide	9	10
Controlled Fine Grind		
#240 Boron Carbide	60	480
#600 Boron Carbide	30	180
#15 Diamond	15	90
#6 Diamond	6	45
Polish		
Rouge	--	--

APPENDIX B

SPECIMEN HISTORY

Material	S/N*	T/N*	Treatment	Yield Modulus, TN/m^2
Silicon	201	1	Rough ground, annealed Destroyed in anneal	---
	202	1	Rough ground, annealed Destroyed in anneal	---
	203	1	Rough ground, partially fine ground (unscheduled), heat treated	0.61
		2	Controlled fine ground, polished	>1.0
	204	1	Rough ground, heat treated	0.35
		2	Diameter etched 120 μ m	>1.0
		3	Controlled fine ground, polished	>1.0
ULE Silica	211	1	Ground, superficially lapped, heat treated. Broken in test	0.10
	212	1	Rough ground, heat treated, diameter etched 200 μ m	>1.0
		2	Diameter etched additional 50 μ m	>1.0
		3	Reground (rough) partially fine ground (unscheduled), heat treated	0.68
		4	Conventional fine ground, heat treated	0.75
		5	Reground (rough), heat treated	0.15

*S/N - Specimen number T/N = Test number

APPENDIX B (Continued)

Material	S/N*	T/N*	Treatment	Yield Modulus, TN/m^2
ULE Silica	212	6	Controlled fine ground	0.95
		7	Polished	>1.0
	213	1	Rough ground, partially fine ground (unscheduled), heat treated	0.39
		2	Heat treated, diameter etched 8 μ m Broken in test	>1.0
	214	1	Rough ground, superficially lapped, heat treated	0.12
		2	Fine ground .083 mm on diameter with #600 boron carbide. Broken in test	0.56
	215	1	Rough ground, heat treated	0.12
		2	Controlled fine ground	0.91
		3	Polished	0.93
CER-VIT 101	221	1	Rough ground, heat treated Broken in mounting	---
	222	1	Rough ground, heat treated Broken in mounting	---
	223	1	Rough ground, partially fine ground (unscheduled) heat treated	0.35
		2	Heat treated, diameter etched 70 μ m Broken in test	>1.0
	224	1	Rough ground, partially fine ground (unscheduled), heat treated	0.38
		2	Heat treated, diameter etched 10 μ m	>1.0

Material	S/N*	T/N*	Treatment	Yield Modulus, TN/m^2
CER-VIT 101	224	3	Heat treated, diameter etched additional 10 μ m	>1.0
		4	Reground (rough), partially fine ground (unscheduled), heat treated	0.80
		5	Conventional fine ground, heat treated	0.83
		6	Reground (rough), heat treated	0.16
		7	Controlled fine ground	1.0
		8	Polished	>1.0
	225	1	Rough ground, superficially fine ground, heat treated	0.23
		2	Conventional fine ground	poor data
		3	Heat treated	0.76
		4	Reground (rough), heat treated	0.20
		5	Controlled fine ground, heat treated	0.99
		6	Polished	>1.0
Silica 7940	231	1	Rough ground, controlled fine ground, polished	1.0
	232	1	Rough ground, controlled fine ground, polished	0.90

APPENDIX C

DELAYED ELASTIC STRAIN EXTRAPOLATION

Determination of End Point

Assumption:

The time history of a test specimen after release of load may be represented closely by the expression

$$A = \frac{B}{t} + K$$

where: A is the total instantaneous strain

B is a factor representing the delayed elastic strain at unity time

t is time after release of load

K is the end point or steady state strain

Let us take time intervals t_1 and t_2 such that

$$t_2 = 10\ t_1$$

then

$$B = t_1(A_1 - K) = t_2(A_2 - K)$$

and

$$K = \frac{\frac{t_2}{t_1} \cdot A_2 - A_1}{\frac{t_2}{t_1} - 1}$$

or

$$K = \frac{10}{9}(A_2 - \frac{1}{10}\ A_1)$$

Determination of Delayed Elastic Modulus

If we take times t of 50 and 100 seconds and subscript the parameters A accordingly, we have

$$A_{50} = \frac{B}{50} + K$$

$$A_{100} = \frac{B}{100} + K$$

$$A_{50} - A_{100} = \frac{B}{50} - \frac{B}{100} = \frac{B}{100}$$

$$B = 100(A_{50} - A_{100})$$

and Delayed Elastic Modulus is found by

$$M_{DE} = \text{stress}/100\cdot(A_{50} - A_{100})$$

APPENDIX D

DATA REDUCTION COMPUTER PROGRAM

```
//CC005337  JCB (NW,
//       ESAB21WX00,10),'W W WCCDS,  773-0729'              8H-H1
//FOR         EXEC PGM=IEYFCRT,PARM=NCMAP,REGION=140K
//SYSPRINT DD SYSOUT=A,UNIT=SYSDA,                                    X
//            DCB=(LRECL=12C,RECFM=FBA,BLKSIZE=1680)
//SYSLIN      DD DSNAME=&LCADSET,DISP=(MCC,PASS),UNIT=SYSDA,
//            DCB=(LRECL=80,BLKSIZE=400),SPACE=(400,(1200))
//SYSIN       DD *
C          REDUCTION CF MICRCYIELD DATA USING  E=E1/T EXTRAPULATION
      INTEGER SAMPLE,GAIN
      REAL SENS(20),ZERC(20),MATL*8(10)
5     READ(5,6) MATL
6     FCRMAT(10A8)
      CALL CALTIT(MATL,SENS,ZERO)
      CALL INVAL(SENS,ZERC)
      READ(5,15) NN
15    FCRMAT(I2)
      GC TC (5,20),NN
20    CONTINUE
      STCP
      END
      SUBRCUTINE CALTIT(MATL,SENS,ZERC)
      REAL MATL*8(10),SENS(20),ZERO(20),LMULT
      INTEGER SAMPLE,GAIN
1C    READ(5,15) MODE,DIAM
2C    WRITE(6,30) (MATL(I),I=1,9),DIAM
15    FCRMAT(I2,8X,F8.0)
3C    FORMAT(1H1,9A8,10X,16HSAMPLE DIAMETER= ,F8.4,7H INCHES /1H0)
      IF(DIAM.GT.C.0) GC TC 60
      WRITE(6,40)
40    FORMAT(45H DIAMETER VALUE IMPRCPER. PROGRAM TERMINATEC. )
      CALL EXIT
60    IF(MCDE.GT.1) GC TC 10C
70    EXMULT=DIAM/0.28
8C    LMULT=(C.28/DIAM)**3
90    GC TC 12C
1CC   EXMULT=1.0
11C   LMULT=(C.28/DIAM)**2
12C   CONTINUE
125   WRITE (6,126) LMULT,EXMULT
126   FORMAT(7H LMULT= ,1PE10.3,10X,7HEXMULT= ,E10.3)
13C   READ(5,140)GAIN,CALP,CALC,CALM,CALV
14C   FCRMAT(I2,8X,4(F8.C,2X))
      IF(GAIN.GT.0) GC TC 150
      WRITE(6,145) GAIN
145   FCRMAT(25H IMPRCPER VALUE CF GAIN =,I6)
      CALL EXIT
15C   IF(GAIN.NE.10)GO TC 210
16C   SENS(1C)=LMULT*CALV/(CALP-CALM)*2.0
17C   ZERC(10)=CALC*SENS(10)
180   WRITE(6,190)SENS(10),ZERO(10)
19C   FORMAT(2CH LCAD SENSITIVITY = ,1PE10.3,14H  LCAD ZERO = ,E10.3)
2CC   GC TC 13C
21C   IF(GAIN.GT.20) GO TO 250
```

```
220    SENS(GAIN)=EXMULT*CALV*2.0/(CALP-CALM)
230    ZERO(GAIN)=CALO*SENS(GAIN)
       WRITE (6,235) GAIN,SENS(GAIN),ZERO(GAIN)
235    FORMAT(13H GAIN NUMBER= ,I4,10X,12HSENSITIVITY= ,1PE11.4,10X,
      15HZERO= ,E11.4)
240    GO TO 130
250    CONTINUE
       RETURN
       END
       SUBROUTINE INVAL(SENS,ZERO)
       REAL SENS(20),ZERO(20),LSTRN,LBEND,LOADV
       INTEGER GAIN
C          COMPUTE SINGLE POINT VALUES BY FORMULA EXTRAPULATION
285    WRITE (6,286)
260    READ(5,270) GAIN,LOADV,ELV,EWV,E1V,EXVM10,EXV
270    FORMAT(I2,8X,6(F8.0,2X))
280    IF(GAIN.GT.20) GO TO 370
286    FORMAT(1H0,20X,41HSINGLE POINT VALUES-FORMULA EXTRAPOLATION/1H0/19
      1X,5HYIELD,6X,4HLOAD,8X,5HLONG.,7X,7HLATERAL/6X,6HSTRESS,6X,6HSTRAI
      2N,6X,6HSTRAIN,6X,7HBENDING,5X,7HBENDING/1H )
290    STRESS=LOADV*SENS(10)-ZERO(10)
300    YIELD= 1.111*SENS(GAIN)*(EXV-0.1*EXVM10)-ZERO(GAIN)
       IF(E1V.EQ.0.0) GO TO 335
310    LBEND=(ELV*SENS(11)-ZERO(11))/12.
320    WBEND=(EWV*SENS(12)-ZERO(12))/12.
330    LSTRN=E1V*SENS(1)-ZERO(1)
       GO TO 340
335    LBEND=0.0
       WBEND=0.0
       LSTRN=0.0
340    WRITE(6,350) STRESS,YIELD, LSTRN,LBEND,WBEND
350    FORMAT(1H ,2X,5(1PE12.3))
360    GO TO 260
370    RETURN
       END
/*
//LKED       EXEC PGM=LINKEDIT,COND=(5,LT),REGION=140K
//SYSPRINT DD SYSOUT=A,UNIT=SYSDA,                                    X
//            DCB=(LRECL=121,RECFM=FBA,BLKSIZE=1573)
//SYSLIB    DD DISP=SHR,DSNAME=SYS1.FORTLIB
//SYSLMOD   DD UNIT=SYSDA,DISP=(,PASS),DSNAME=&&PDS(MEMB),
//               SPACE=(1024,(200,10,1),RLSE),DCB=BLKSIZE=1024
//SYSUT1    DD UNIT=SYSDA,SPACE=(3250,(250,5),RLSE),DCB=BLKSIZE=1024
//SYSLIN    DD DSNAME=&LOADSET,DISP=(OLD,DELETE)
//GO        EXEC PGM=*.LKED.SYSLMOD,COND=(5,LT,LKED),REGION=140K
//FT05F001 DD DDNAME=SYSIN
//FT06F001 DD SYSOUT=A,UNIT=SYSDA,                                    X
//            DCB=(LRECL=130,RECFM=FBA,BLKSIZE=1430)
//FT08F001 DD DSNAME=&&RW,DISP=(NEW,DELETE),UNIT=SYSDA,               X
//            DCB=(LRECL=80,RECFM=FB,BLKSIZE=400),SPACE=(400,1)
//SYSIN     DD *
 DATE 4/4/72 ULE 211-1 AS GROUND
01          0.301
10          0.9955     -0.0009    -0.9958    91.5
```

11	0.0180	-0.0028	-0.0246	1.00E-05		
12	0.0177	-0.0032	-0.0245	1.00E-05		
01	0.0200	-0.0008	-0.0219	1.00E-05		
04	1.9951	0.0000	-1.9837	1.00E-06		
30						
04	0.0106	-0.0036	-0.0024	0.0820	0.0139	0.0067
04	-0.0214	0.0000	-0.0048	-0.1226	-0.0072	0.0008
04	0.0258	-0.0028	-0.0011	0.1676	0.0162	0.0056
04	-0.0492	0.0000	-0.0072	-0.2842	-0.0091	0.0017
04	0.0672	-0.0062	0.0015	0.4102	0.0268	0.0130
04	-0.1045	0.0000	-0.0131	-0.6042	-0.0262	-0.0023
04	0.1511	-0.0022	0.0061	0.8860	0.0586	0.0380
04	-0.2692	0.0000	-0.0315	-1.5430	-0.1103	-0.0658
04	0.4189	0.0000	0.0055	2.0100	0.4434	0.3289
30						
02						

APPENDIX E

ADJUSTMENT OF NEGATIVE YIELD MODULUS TO TEST DIAMETER

Assume that the "negative yield" angular deflection per unit length (α) is a function only of torque (T) for a given material.

Thus

$$\alpha = K\ T$$

The apparent specimen strain (ε) is found by

$$\varepsilon = \frac{1}{2}\, d\alpha$$

where d is the specimen test section diameter.

The stress (S) exerted upon the test section by the torque T is

$$S = \frac{16T}{\pi d^3}$$

so that the "Modulus" of negative yield M is

$$M = \varepsilon / S = \frac{\pi d^3}{16\ T} \cdot \frac{d\alpha}{2} = \frac{\pi d^4 \alpha}{32\ T}$$

The "modulus" so defined is obviously a function of diameter. If we have the modulus defined at a particular diameter d_o and wish to determine the effective apparent negative yield strain at a different diameter d_1, then we have

$$M_1 = M_o\ (d_o/d_1)^4$$

and

$$\varepsilon_1 = \frac{S}{M_o\ \ (d_o/d_1)^4}$$

APPENDIX F

NORMALIZATION OF YIELD MODULUS TO CONSTANT DIAMETER

The yield of the brittle materials measured in this program is entirely dependent upon surface condition, and therefore may be assumed to lie within a thin layer on the outer surface of the test section of the specimen. A convenient model assumes small displacement centers of large local yield scattered over the surface of the cylindrical test section, producing an effective average yield strain in the surface. This yield strain, if unrestrained, would result in an angular deflection per unit length (α_1). The body of the rod, however, has not undergone yield and resists the angular deflection imposed by the surface, with a resultant angular deflection per unit length (α_2) less than α_1.

Let us assume the following parameters:

α_1 = unrestrained surface yield torsional deflection per unit length

α_2 = equilibrium torsional deflection per unit length of body and surface

J_1 = axial moment of inertia of yielded surface

J_2 = axial moment of inertia of unyielded body

T_1 = elastic torque of surface in equilibrium with body

T_2 = elastic torque of body in equilibrium with surface

δ = thickness of disturbed (yielded) surface layer

d = diameter of body

G = modulus of rigidity.

The polar moments of inertia are:

$$J_1 = \frac{\pi d^4 - \pi(d - 2\delta)^4}{32}$$

$$J_2 = \frac{\pi(d - \delta)^4}{32}$$

If we assume the thickness (δ) of the disturbed surface to be small with respect to the overall diameter, the expressions for moment of inertia reduce to

$$J_1 = 1/4\ \pi\delta d^3$$

$$J_2 = 1/32\ \pi\ d^4$$

The elastic torques exerted upon the two portions of the rod in equilibrium are equal, and expressed by:

$$T_1 = G\ J_1(\alpha_1-\alpha_2) = 1/4\ G\ (\alpha_1-\alpha_2)\pi\delta d^3$$

and

$$T_2 = G\ J_2\ \alpha_2 = 1/32\ G\ \alpha_2\pi d^4$$

thus

$$\alpha_2 = 8\ \alpha_1\delta/(8\delta+\ d)$$

If we again assume that

$$\delta \ll d,$$

we may make the approximation

$$\alpha_2 = 8\ \alpha_1\ \delta/d$$

Let

y_1 = yield strain of surface layer

y_2 = apparent yield strain of combined surface and body

then

$$y_1 = d\ \alpha_1/2$$

$$y_2 = d\ \alpha_2/2$$

and

$$y_2 = 8\ y_1\ \delta/d$$

For a given surface yield, this indicates the apparent yield to vary inversely as the test section diameter.

The pseudo-modulus M_y used in this report to compare materials and surface conditions may be related to specimen diameter and surface strain by

$$M_y = \text{stress}/(y_2)^{1/2}$$

or

$$M_y = \text{stress} \cdot (d/(8\ y_1\delta))^{1/2}$$

which implies that the observed modulus of yield is proportional to the square root of the specimen test section diameter. To normalize the results to a given diameter d_o, the operation indicated by

$$M_y\ (\text{normalized}) = M_y\ (\text{apparent}) \cdot (d_o/d)^{1/2}$$

must be performed.

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