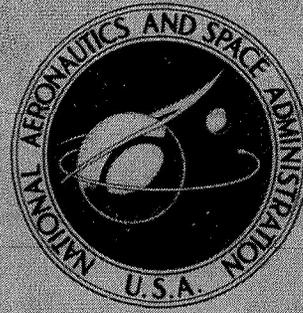


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**ABSOLUTE CAPACITANCE MICROCREEP
AND DIMENSIONAL STABILITY
MEASURING SYSTEM**

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ABSOLUTE CAPACITANCE MICROCREEP AND DIMENSIONAL STABILITY MEASURING SYSTEM

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SUMMARY

An absolute capacitance microcreep and dimensional stability measuring system is described. The system individually measures and records on a printer as many as 100 microcreep or dimensional stability measurements on a consecutive sampling basis, and can be used over a temperature range from absolute zero to about 300°F. The salient features of the system are its long time stability and strain sensitivities (10^{-9} in. or less) that can be achieved. Microcreep and dimensional stability data for 1000 hour durations are presented on 400C steel and Beryllium I 400; and Free Cut Invar, Titanium 6 AL-4V, and Ferrotic MS-5, respectively.

INTRODUCTION

The scarcity of microcreep properties of materials is probably due in great part to the lack of long time (greater than 30 days) stability and sensitivity of the microcreep displacement sensor; and the difficulty, and inconvenience of making periodic high precision measurements over long periods of time. For proper design of precision instruments, however, the micromechanical properties of materials are requisite because permanent dimensional changes of 1×10^{-6} in. can have deleterious results on instrument performance and reliability. Moreover, the creep and dimensional stability characteristics of materials are required over temperatures ranging from cryogenic to 300°F which is the possible temperature excursion for most precision instruments in use today for guidance and navigation purposes. With the technique to be described, the recoverable and non-recoverable dimensional changes resulting from (1) short and long application of stress, and (2) metallurgical (thermodynamic) instabilities and relaxation of residual stresses can be measured at strain sensitivities of the order of 10^{-9} in. (or less sensitivity) over large temperature ranges with a print out of data (or recorded on punch cards for further computer data processing) at selected periodic intervals.

ABSOLUTE CAPACITANCE MEASUREMENT

The use of absolute capacitance measurements to determine length changes in microcreep studies was first suggested by Rutherford and Hughes*. With a capacitance extensometer wired for three terminal connections to the hand operated General Radio Type 1620-A Capacitance Measuring Assembly (ref. 1) highly

*Personal communication

accurate and sensitive capacitance (displacement) measurements are possible. The 1620 Assembly has a precision of 0.01 percent at one picofarad (pF) and a stability with respect to time and temperature better than 0.01 percent per year in normal use. With a capacitance (gauge) plate area of about one square inch, for example, it is possible to determine displacements to a sensitivity of 10^{-7} in. at 100 pF, 10^{-8} in. at 200 pF and greater sensitivities at higher capacitance values.

If an automatic capacitance measuring system consisting of an automatic capacitance bridge, a scanner (switch), data printer, and timer is used in place of the hand operated bridge it is possible to continuously sample and record up to 50 or more stations at selected time intervals with comparable precision of the hand bridge. The General Radio 2990-9167 Automatic Capacitance Measuring System (ref. 1) is presently being used to measure and record room temperature microcreep properties of materials currently being used in gyroscopes and accelerometers. Measurements at low (cryogenic) and intermediate temperatures (300°F) have not been made yet, but no difficulties are contemplated other than the constancy of temperature required if detection of very small displacements is desired.

The essential features of the Automatic Microcreep measuring system are shown in Figures 1a and 1b. The main construction points of the capacitance gauge sensor are shown in Figure 2. Referring to the block diagram Figure 1a, one or as many as 50 (channels) specimens can be monitored by the scanner switch on a consecutive selection basis. Operation of the system is started by the timer which actuates the scanner control. This control selects a channel at the scanner switch which is then measured by the automatic bridge and the results printed by the printer on a signal from the bridge when it is balanced at the correct capacitance value. When the data print is completed, the scanner control switches to the next channel and the process is repeated for as many channels that are being monitored.

The capacitance gauge shown in Figure 2 differs from the relative capacitance gauges described by Brown (ref. 2) and Roberts et al. (ref. 3) in the three terminal wiring of the capacitance plates. Other features are, the gauge is made entirely of invar, heat treated for maximum dimensional stability and the capacitance plates are bonded to the gauge base with a thin layer of a low expansion dielectric adhesive (Tra-Bond BA 2153).

STRAIN CALIBRATION

The experimental calibration curve Figure 3 was obtained by mounting the capacitance gauge on a specimen (cut in center of

gauge length) adapted for use in a high magnification extensometer calibration micrometer (Instron Type G55-1, readable to 5×10^{-6} in.) and reading the absolute capacitance values for increased settings (openings) on the micrometer. The minimum zero gap was arbitrarily selected as the first reading that could be obtained after breaking electrical contact of the two capacitance plates by opening the micrometer. Both the hand operated G. R. Type 1620 (0.01 percent accurate) and the automatic G. R. Type 1680 (0.1 percent accurate) capacitance measuring assemblies were used on successive runs in a constant temperature ($68 \pm 1/2^\circ\text{F}$) and humidity 42 ± 3 percent) room, Figure 4, to establish the capacitance versus gap calibration curve shown in Figure 3. Both capacitance assemblies yielded the same curve indicating the high degree of accuracy, repeatability, and stability with this measuring technique.

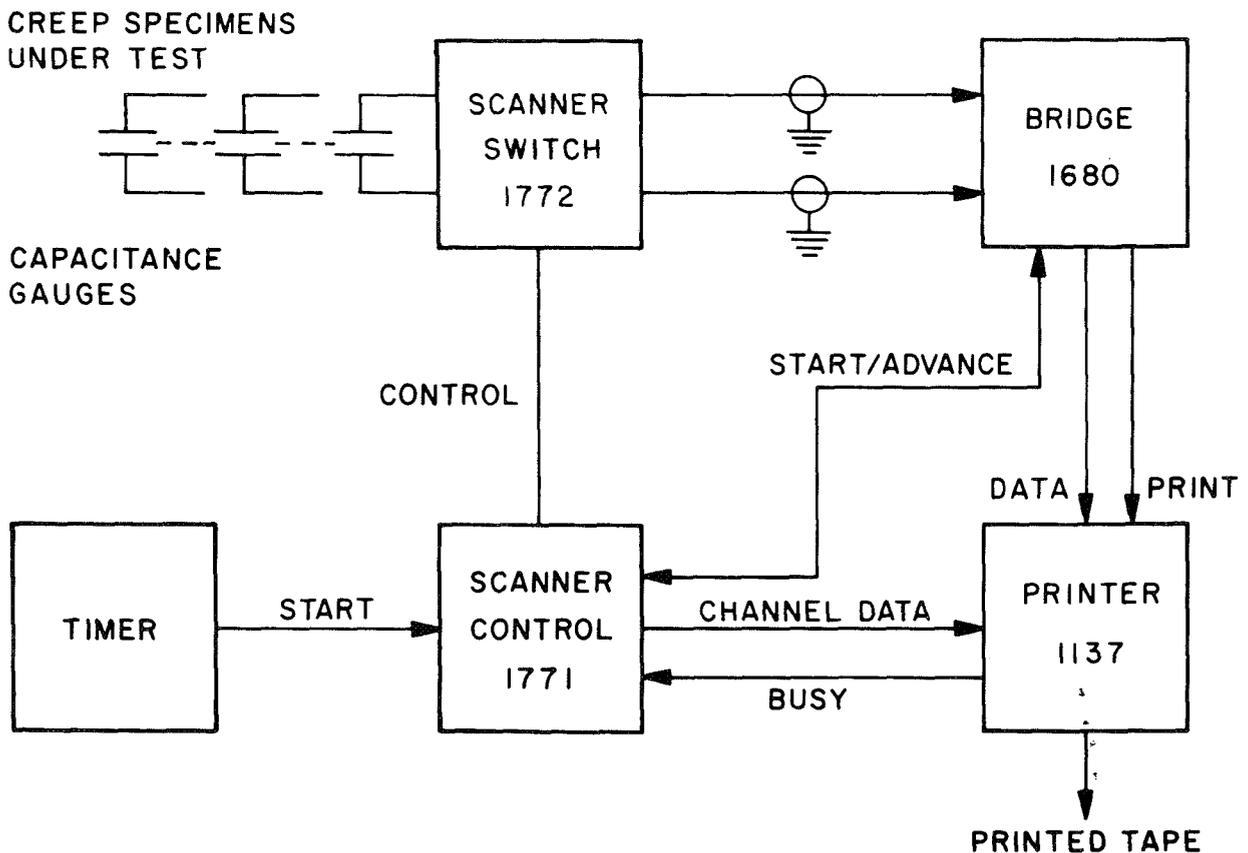


Figure 1a.- Simplified block diagram of automatic microcreep measuring system

The calculated values of the gap were determined also using the relationship $C = \frac{.225 A}{G}$, the standard capacitance equation, where C = capacitance in picofarads (pF), A = area of one gauge plate in square inches, and G is the distance in inches between the plates. If the calculated gap values are then compared with the measured values for a given capacitance, as shown in Table I, the difference is constant at about 0.74 mil or 744 microinches for

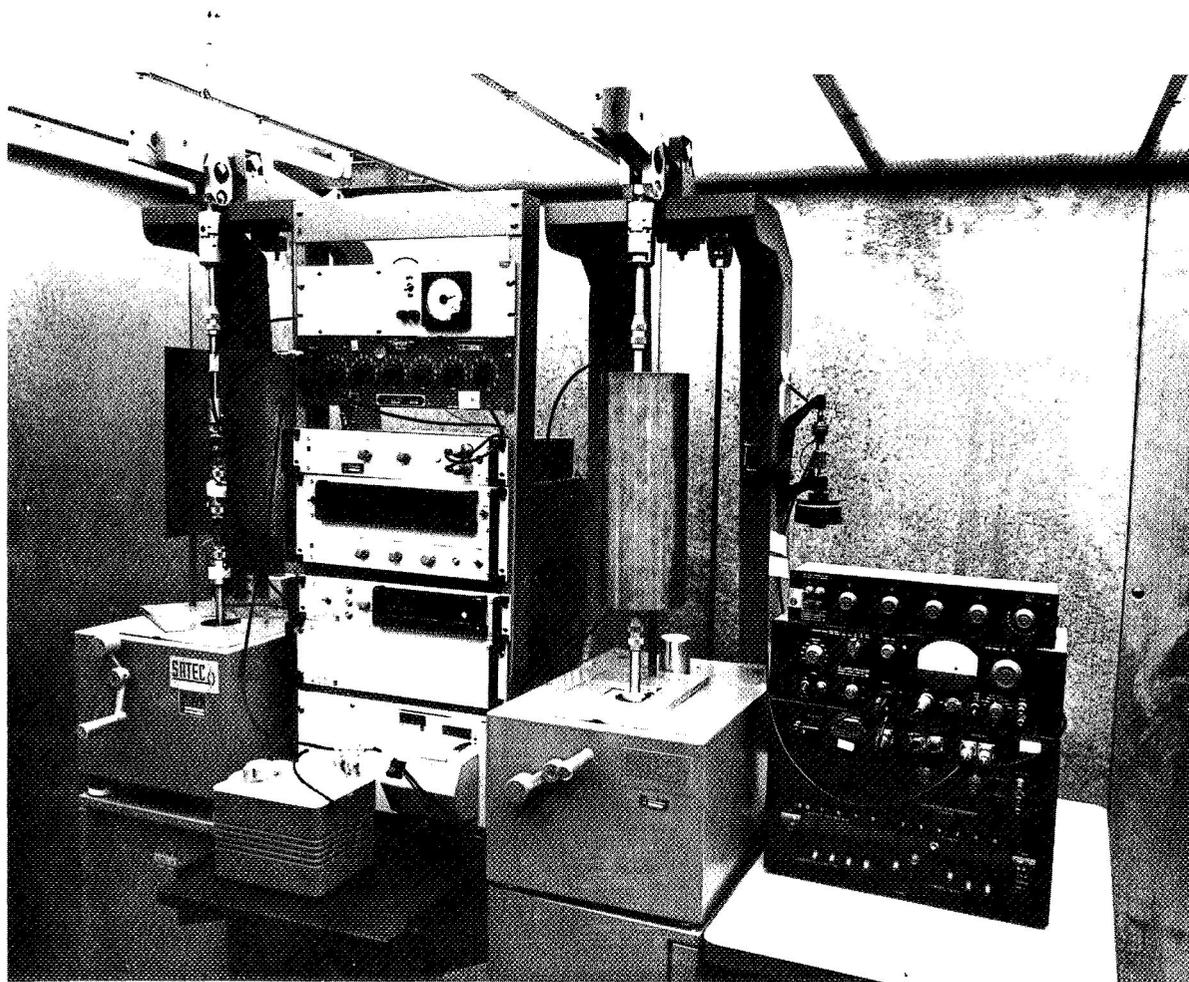


Figure 1b.- Automatic capacitance measuring system between two creep frames and capacitance hand bridge at right

all capacitance values. Also the difference between the measured and calculated gaps for any two given capacitance values is essentially equal. Over the total range from 160 pF to 50 pF, which to date constitutes most of the workable range for constant room temperature creep studies, the measured and calculated gap

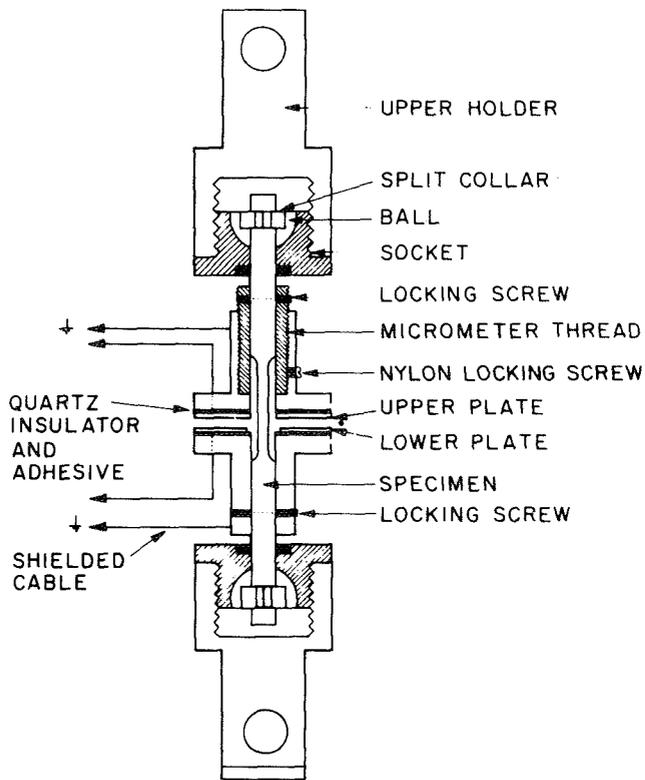
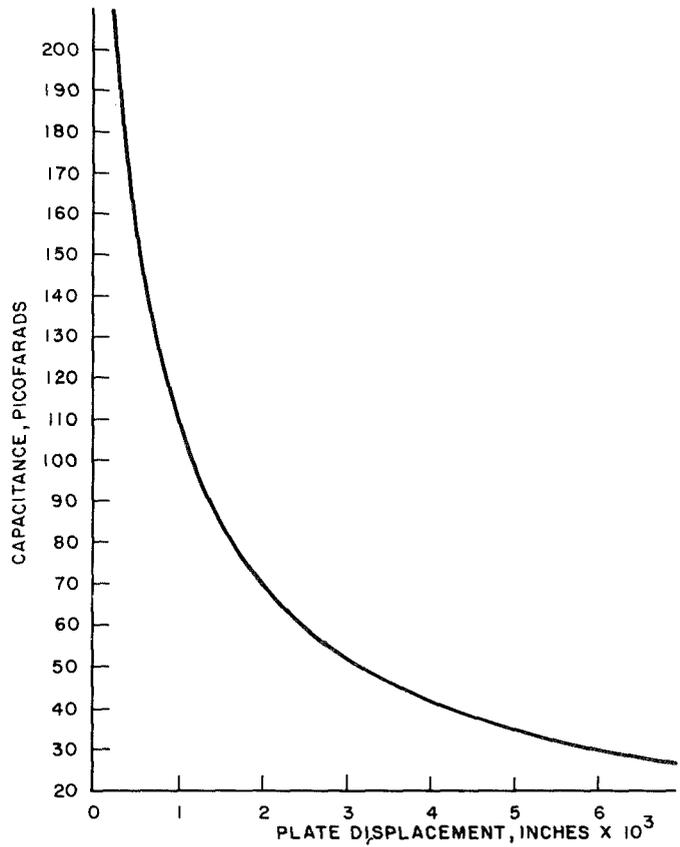


Figure 2.- Capacitance gauge assembly showing specimen, three terminal wiring connection and grips

Figure 3.- Strain calibration curve showing capacitance versus displacement



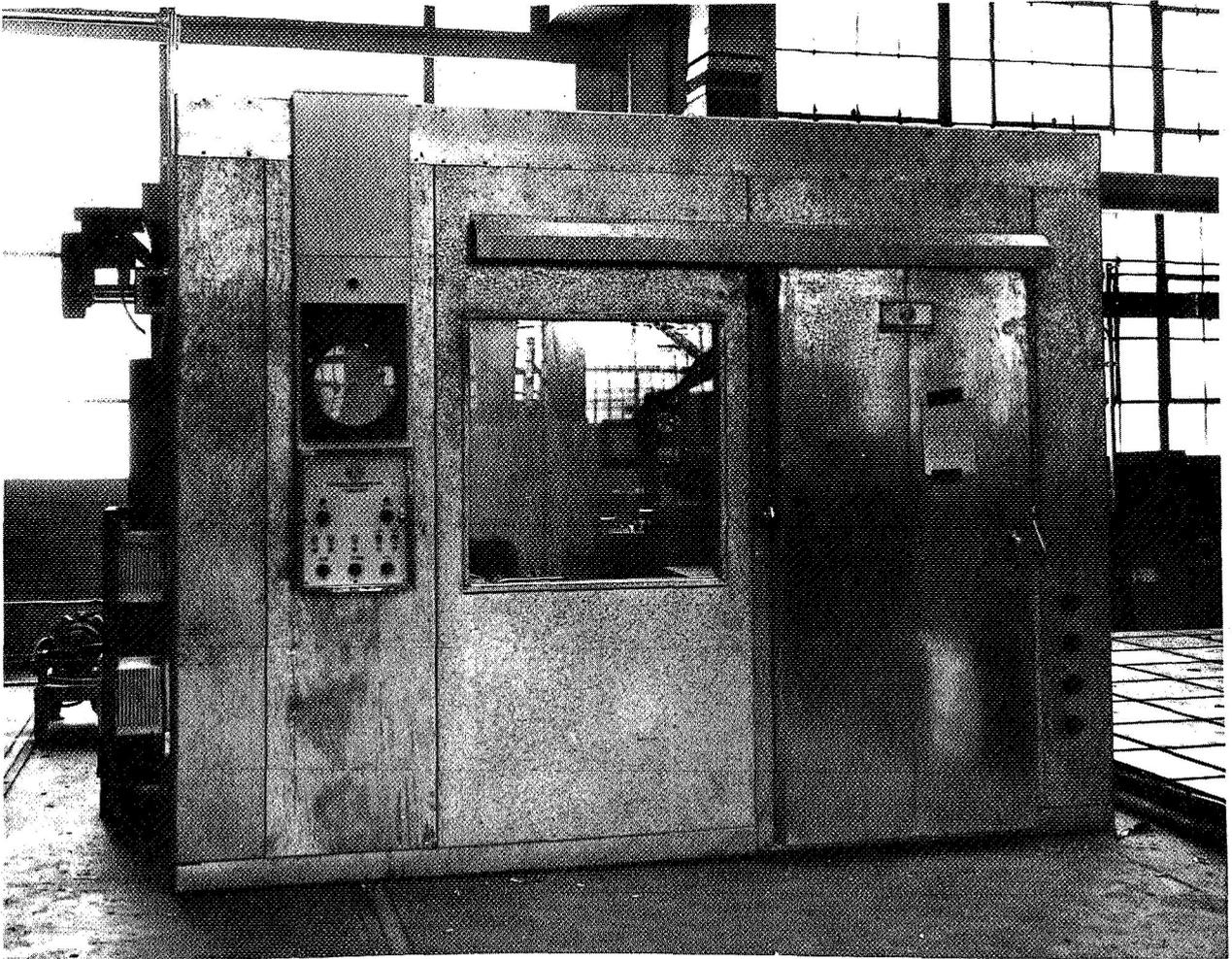


Figure 4.- Constant temperature and humidity room

differences are in error by about 0.1 percent. For 10 pF intervals, the measured and calculated gap differences disagree by about 3.22 percent (average) as shown in Table II. Considering that the measured values will agree more closely with calculated values as more stable gauges, parallel plates, improved micro-meters and stable temperatures are developed, creep extension can be determined rather accurately by calculating the gap or displacement from the measured capacitance values rather than using the calibration curve exclusively. In fact, all creep loading and unloading characteristics reported henceforth will be differences of calculated gap values using the relationship

$$\Delta G = .225 A \frac{1}{C_2} - \frac{1}{C_1}$$

where C_2 equals the lower (final) and C_1 (initial) capacitance values, respectively.

TABLE I.- COMPARISON OF MEASURED AND CALCULATED GAP DISPLACEMENTS

PICO FARAD	MEASURED (CALIBRATION), IN.	CALCULATED, IN.	DIFFERENCE	% DIFFERENCE*
160	0.000475	0.001203	728	+1.33
150	0.000550	0.001283	733	+0.86
140	0.000630	0.001375	745	+0.07
130	0.000740	0.001481	741	+1.35
120	0.000860	0.001604	744	0
110	0.000990	0.001750	760	-0.91
100	0.001160	0.001925	765	-1.1
90	0.001380	0.002139	759	-0.70
80	0.001660	0.002406	746	-0.08
70	0.002000	0.002750	750	-0.22
60	0.002450	0.003208	758	-0.44
50	0.003150	0.003850	700	+1.14
			SUM = 8929	+1.16
			K = AVG 744	+0.086

$$* G \frac{\text{Measured} + K - \text{Calculated}}{\text{Calculated}} \times 100$$

$$G = \frac{0.192482}{C}$$

TABLE II.- COMPARISON OF DIFFERENCES BETWEEN MEASURED
AND CALCULATED GAP DISPLACEMENTS

CAPACITANCE INTERVAL, PF	MEASURED INTERVAL, IN. x 10 ⁻⁶	CALCULATED INTERVAL, IN. x 10 ⁻⁶	DIFFERENCE IN. x 10 ⁻⁶	% DIFFERENCE*
100-150	75	80.2	+ 5.2	- 9.73
150-140	80	91.7	+11.7	-15.60
140-130	110	105.7	- 4.3	+ 1.67
130-120	120	123.4	+ 3.4	- 4.81
120-110	130	145.8	+15.8	-12.57
110-100	170	175	+ 5.0	- 4.3
100- 90	220	213.9	- 6.1	+ 1.66
90- 80	280	267.3	-12.7	+ 3.80
80- 70	340	343.7	+ 3.7	- 1.82
70- 60	450	458.3	+ 8.3	- 2.37
60- 50	700	642	-58	+ 8.64
			SUM =	35.43
			K = AVG	- 3.22

* $\frac{\text{Measured} + K - \text{Calculated}}{\text{Calculated}} \times 100$

$$G = \frac{0.192482}{C}$$

Likewise, the strain sensitivity for a given capacitance is given by

$$\frac{dC}{dG} = \frac{0.225 A}{g^2} = (\text{pF}/10^{-6} \text{ in.}) \quad \text{or} \quad \frac{dG}{dC} = 10^{-6} \text{ in/pF}$$

where G is the calculated gap, or the measured gap from Figure 3 plus the constant 744.0×10^{-6} in. Again, the calculated sensitivity is believed to be more realistic than that measured from the slope of calibration curve for the reasons previously mentioned.

STRAIN RESOLUTION AND SENSITIVITY

The automatic capacitance bridge has a maximum resolution of 0.01 pF and a repeatability of ± 0.02 pF in range 1 (150 pF MAX) at 1000 Hz. The bridge also can be used to indicate the difference between two capacitors by connecting a fixed capacitor to the external standard terminal on the rear of the bridge. This means that the nominal value of a capacitor (such as the capacitance value of a creep specimen under test) can be balanced out by an external precision capacitance standard to indicate changes in the creep capacitor as small as the resolution of the bridge (0.01 pF). This also means that 0.01 resolution can be obtained for capacitance values greater than 150 pF, the upper limit for range 1. For example, a 300 pF reading on a creep extensometer with a plate area of about 0.88 in.² has a sensitivity of 2.2×10^{-6} in. per picofarad. In range two of the bridge (150 - 1500 pF) the resolution is ± 0.1 pF or about $\pm 0.22 \times 10^{-6}$ in. By using a stable precision external standard such that the difference of the creep gauge value and the external standard is in range 1 (0.01 pF - 150. pF) the resolution is increased to 0.01 pF or in this case, 0.022×10^{-6} in.

Greater sensitivity and resolution can be achieved by using a precision decade transformer in conjunction with a precision external standard. A Gertz decade autotransformer adjusted for a gain of 10 has been put in the line between the creep capacitance gauge and the bridge with a precision external standard connected to the bridge. Again, assume a capacitance gauge with an area of 0.88 square inch and a typical capacitance value of 100 picofarads. The sensitivity $\frac{dc}{dg}$ of this combination is 0.505 per micro-inch or 19.78 microinch per picofarad. Without magnification and subtraction the best resolution in range 1 is 0.01 pF or 0.1978×10^{-6} inch and a repeatability of about $\pm 0.4 \times 10^{-6}$ in. If the capacitance is multiplied ten times by the transformer, 1 pF now equals 1.978 microinch. By subtracting a sufficient

capacitance to bring the bridge back to range 1 it is now possible to read to 0.01 pF or 0.01978×10^{-6} in. with a repeatability of about $\pm 0.04 \times 10^{-6}$ inch. With a capacitance value 300 pF on a creep gauge, detection of changes in extension or compression of $\pm 2.2 \times 10^{-9}$ in. ($1A = 3.937 \times 10^{-9}$ in.) appear feasible provided the temperature, gauge, bridge, external standard, and transformer all possess the requisite stability.

MATERIALS AND PROCEDURES

To demonstrate the stability, sensitivity, and repeatability of the system, 1000 hour long microcreep tests were carried out on Beryllium I 400 and hardened 440 C steel; dimensional stability tests were carried out over the same time duration on Free Cut Invar 36, Titanium 6AL-4V and Ferrotic MS-5, a sintered carbide material.

The 440 C steel specimens were machined from 0.75 in. diameter bar stock supplied by the M.I.T. Instrumentation Lab. from materials used in a recent bearing development Contract NAS 9-3079. Ferrotic MS-5 was purchased from Sintereast Corp. as round bars 6 inches long, 0.5 in. in diameter. The BeI 400 specimens were purchased finished machined, and heat treated, from the Brush Beryllium Co. who reported that 2 to 3 mils were removed by etching followed by 1 to 2 mils stock removal by machining and polishing to size. The Titanium 6AL-4V specimens were machined from 0.5 in. diameter bar stock and tested in the finished machined state without any subsequent heat treatment.

The chemical composition heat treatments and physical properties of the materials are shown in Tables III, IV, and V, respectively. All specimens with the exception of the Invar were machined to 0.375 in. round tensile specimens with 0.125 in. gauge length diameters and 0.5 in. gauge lengths. All specimens were 4 inches long. The Invar specimen was a 0.375 in. round about 7 inches long with no reduced gauge section. All heat treated specimens were rough machined about 15 mils oversize, heat treated, and finished machined. The 440 C specimens were stress relieved after finish machining.

All tests were made in the constant temperature and humidity room shown in Figure 4. About half-way through the evaluation, it was determined that the room temperature recorder had about a 0.5°F dead band which precluded any meaningful data normalizing up to that time. After the recorder was adjusted and working properly, it was determined that the room held the temperature at about $69.25^{\circ}\text{F} \pm 0.3^{\circ}\text{F}$ for considerable lengths of time but that occasionally changes as great as 1°F could occur before the control system reacted to bring the temperature back to the

TABLE III.- CHEMICAL COMPOSITIONS (WT. %)

MATERIAL	C	Mn	Si	Cr	Ni	Se	S	P	Mo	Al
FREE CUT INVAR 36*	0.12	0.90	0.35	-	36.0	0.20	-	-	-	-
440C L ¹	1.0	0.37	0.38	16.5	0.18	-	0.006	0.012	0.46	-
BeI400L ²	0.20	0.01	0.02	-	-	-	-	-	-	0.02
Ti * 6 AL-4V	0.1	-	-	-	-	-	-	-	-	6.0
FERRO- * TIC MS-5	-	-	-	9.3	4.0	-	-	-	2.7	-

MATERIAL	Mg	Co	Ti	TiC	Va	N	Fe	Other	BeO	Be
FREE CUT INVAR 36*	-	-	-	-	-	-	BAL	-	-	-
440C L ¹	-	-	-	-	-	-	BAL	-	-	-
BeI400L ²	0.01	-	-	-	-	-	0.12	0.10	5.52	BAL
Ti * 6 AL-4V	-	-	BAL	-	4.0	0.03	-	-	-	-
FERRO- * TIC MS-5	-	3.3	1.0	33.6	-	-	46.1	-	-	-

* NOMINAL

1 Carpenter Steel Co. Z 81849-2 Bar IA

2 Brush Beryllium Co. Lot. No. 5114 Grain Size: 9 Microns

TABLE IV.- HEAT TREATMENTS

Alloy	
Free Cut Invar 36 440C ¹	1500°F - 30 Min.; H2O quench; 600°F-1HR, Air Cool 1900°F - 30 MIN. Rapid Oil Quench at 100°F followed by 50 MIN subcool in gas at -100°F and 30 MIN subcool at -320°F. Temper 2 hrs at 300°F followed by same subcool treatment. Retempering at 300°F for 2 hrs. Stress relieved (after finished machining) at 300°F for 5 hrs.
BeI400 ²	1450°F - 1 hr. followed by a cooling rate of 100°F per hour in protective atmosphere.
Ferro-Tic MS-5	1800°F - 30 MIN, air cool. Aged at 900°F 12 hrs.

¹At M.I.T. Instrumentation Lab.

²At Brush Beryllium Co.

TABLE V.- PHYSICAL AND MECHANICAL PROPERTIES*

Material	E, PSI x 10 ⁶	Hard- ness Rockwell	Expansion Coef in/in/°F x 10 ⁻⁶
Free Cut Invar 36	20.5	B-70	0.89
440C	29.0	C-61.7	5.7
BeI400	42.4	-	6.28
Ti 6AL-4V	15.8	-	4.8
Ferrotic MS-5	42.0	C64	3.4

* Nominal

69.25°F average. When data was normalized, the average of 4 readings at 6 hour intervals was used as the average daily temperature. The relative humidity remained constant at about 43 percent \pm 3 percent during the tests.

In dimensional stability measurements the total length changes were figured over a gauge length of 1.8 inches which is the average distance between the shoulder locking screws on the upper and lower gauge bodies, Figure 2. When microcreep was evident, the creep strain was assumed to have occurred in the 0.5 in. gauge length only.

At the end of a given creep test, the specimens were unloaded in an attempt to measure the recoverable elastic strain. In most cases the results were unsatisfactory unless the lower pull rod was completely removed.

The repeatability of the overall system was determined by hourly monitoring a General Radio 100 PF standard (0.2 percent accuracy) in excess of 45 days. The repeatability was determined to be at least 0.1 percent which is the specification for the system.

The capacitance of each test specimen was recorded on tape at 65 minute intervals (about 22 readings daily) during the tests. Occasionally the printer failed, and single readings were taken manually, but not hourly, when the occasion warranted.

To best show the overall stability and repeatability, it was decided to plot values on a daily basis over the 42 day (1000 hour) test period. In Figures 5A, and 6, each point represents a single (8AM) reading. In Figures 5B, 7, 8 and 9 each point represents the average of 22 readings normalized to 69.2°F.

Creep tests on Be and 440C steel were carried out with two specimens loaded in tandem. Except in the case of the BeI400 at 10,000 psi, one gauge failed to work properly in each case due primarily to faulty wiring. It was decided to continue the tests rather than unload and correct the trouble. The initial setup and gauge setting was used for BeI400 tests at 2000, 4000, 6000, and 8000 psi tests by just carefully increasing the load on the lever arm without disassembling.

Dimensional stability measurements were made with the same type of gauge as that used in creep tests. After the gauges were mounted and the gap set, the specimens were placed horizontally and supported at their ends in V grooved fixture with no constraint.

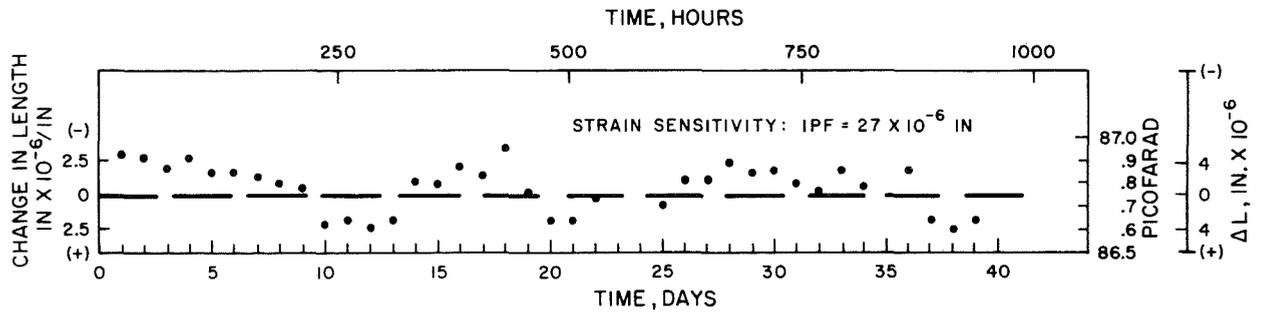


Figure 5A.- Microcreep test on 440C steel RC 62; 32,600 psi at room temperature

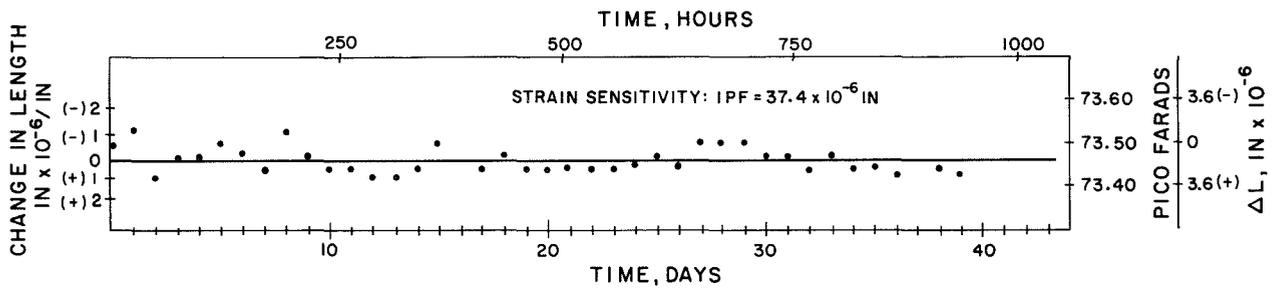


Figure 5B.- Microcreep test on 440C steel, RC 62; 82,000 psi

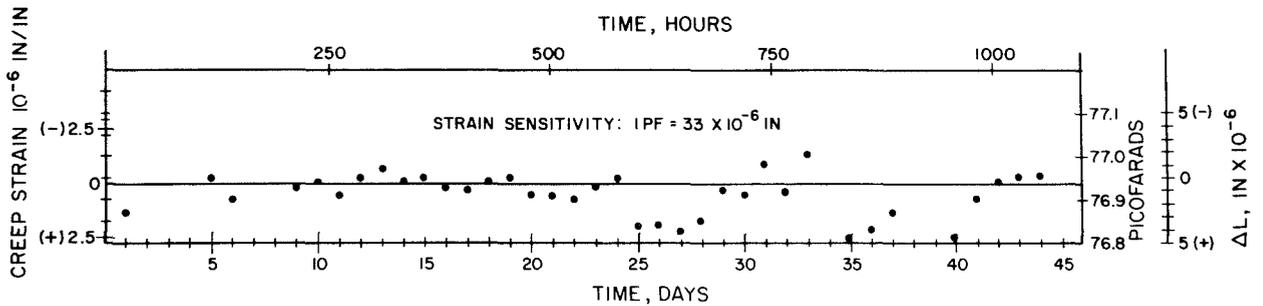


Figure 6.- Microcreep test on I400 Be, at room temperature and 8000 psi (\approx 63% mys)

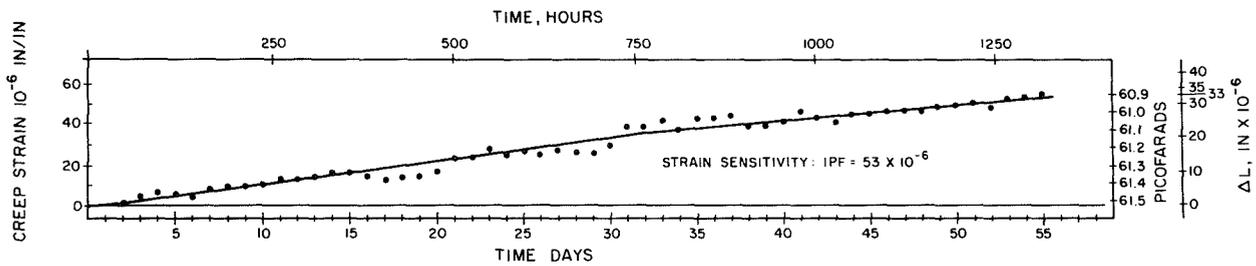


Figure 7.- Microcreep test on I400 Be, at room temperature and 10,000 psi ($\approx 79\%$ mys)

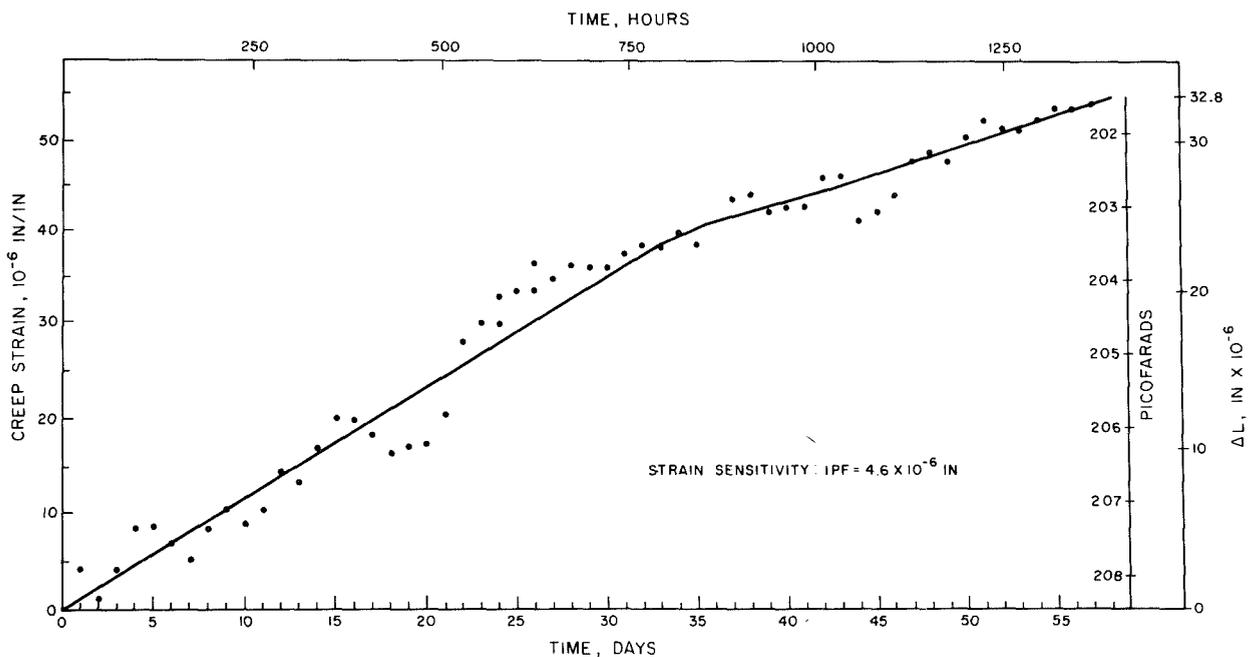


Figure 8.- Microcreep test on I400 Be, at room temperature and 10,000 psi ($\approx 79\%$ mys)

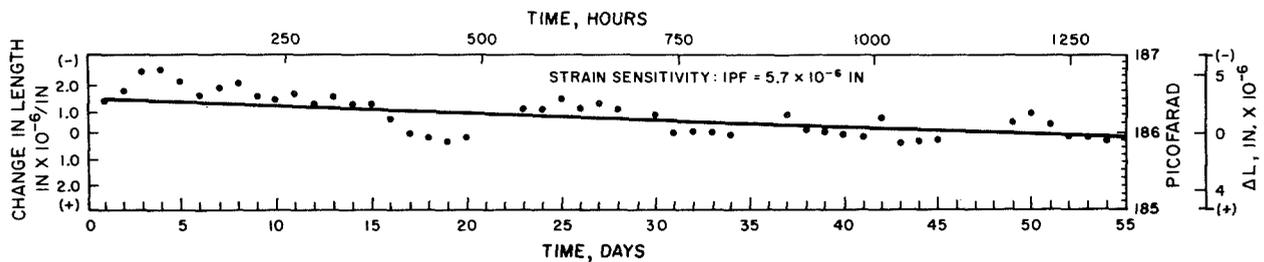


Figure 9.- Dimensional stability of freecut invar (15 min. at 1500°F , water quenched; 1 hr. at 600°F , aircooled)

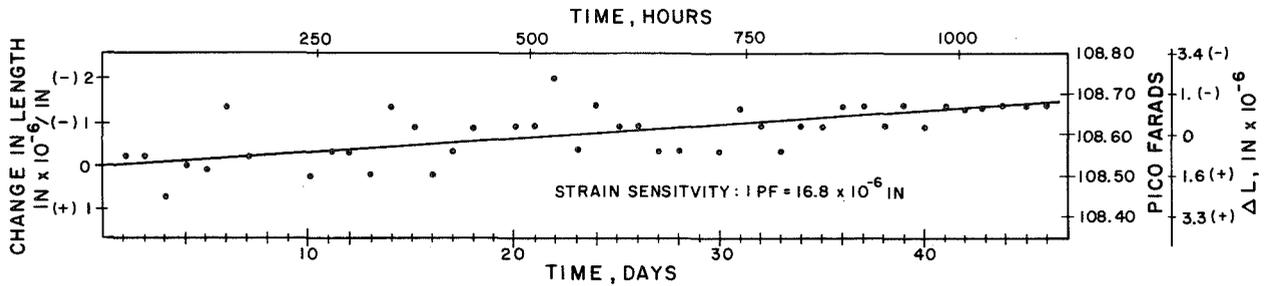


Figure 10.- Dimensional stability of titanium 6AL-4V

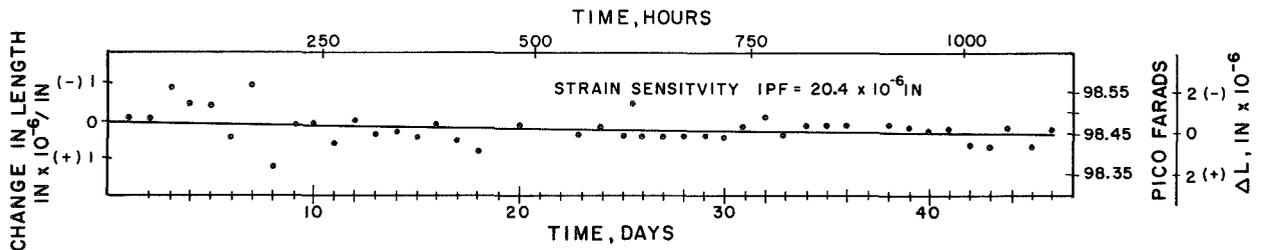


Figure 11.- Dimensional stability of Ferrotic MS-5

RESULTS

Microcreep

The results of room temperature creep tests on hardened and tempered 440C steel at stresses of 32,600 and 82,000 psi are shown in Figures 5A and 5B, respectively. No creep is evident in either case, and the scatter of the points is within the length changes expected for $\pm 0.3^\circ\text{F}$ temperature variations.

Figure 6 shows the results on BeI400 tested at 8000 psi and no creep is discernable. This specimen was also tested for 1000 hour intervals at stresses of 2000, 4000 and 6000 psi with no creep extension. The microcreep results on the same Be specimens tested at 10,000 psi are shown in Figures 7 and 8. Each specimen has crept about 55 microinches in the 55 day test period. These specimens were loaded in tandem with the gauge on one set for a low (61 pF) sensitivity, Figure 7, and the gauge on the other set for a high (205 pF) sensitivity, Figure 8. The capacitance from the high sensitivity gauge was magnified ten times in a Gertsch auto transformer before being measured by the capacitance bridge.

To bring the capacitance value within range 1 on the bridge, a decade capacitance standard was used to subtract a constant capacitance of 1951 pF. Thus, the bridge measured 99 pF to two decimal places (0.01 pF). If the bridge read 98.62 for example, and the constant 1951 is added, the result is 2049.62 which

constitutes reading the 205 pF to three places to the right of the decimal. Since at 205 pF a change of 1 pF corresponds to a calculated change in length of 4.6×10^{-6} in. for the gauge used, it is feasible that changes in length of 4.6×10^{-9} in. can be detected. Long time repeatability tests on the multiplication factor and the capacitance standard are being carried out to assess the possible overall accuracy of this technique. Precision length measurements of creep specimens using an electronic comparator are also planned but changes in length can be read to about 10^{-7} in. at best with this approach. In Figure 8, it is estimated that changes in length were measured to 10^{-7} in. sensitivity, but since temperature changes of 0.1°F amount to about 1×10^{-6} in. change in the 1.8 in. gauge length, considerable fluctuation of the data points is observed.

Dimensional Stability

Free Cut Invar, Figure 9, indicates than an expansion of about 1.5×10^{-6} in./in. occurred in a 55 day period. Since the capacitance gauge was made from the same Invar material and similarly heat treated, it was felt that little, if any, change should be noted in the dimensional stability of this material. In view of the fluctuations in the data points this test is being continued to measure and investigate this behavior more thoroughly.

Titanium 6AL-4V, Figure 10, shows a contraction of about 1.5×10^{-6} in./in. and Ferrotic MS-5, Figure 11, a possible slight expansion in a 45 day period. The scatter of the data points suggests that better temperature monitoring and normalizing procedures be carried out to see if the scatter can be reduced.

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