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Designation: E 289 – 04

Standard Test Method for Linear Thermal Expansion of Rigid Solids with Interferometry¹

This standard is issued under the fixed designation E 289; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of linear thermal expansion of rigid solids using either a Michelson or Fizeau interferometer.

1.2 For this purpose, a rigid solid is defined as a material which, at test temperature and under the stresses imposed by instrumentation, has a negligible creep, insofar as significantly affecting the precision of thermal length change measurements.

1.3 It is recognized that many rigid solids require detailed preconditioning and specific thermal test schedules for correct evaluation of linear thermal expansion behavior for certain material applications. Since a general method of test cannot cover all specific requirements, details of this nature should be discussed in the particular material specifications.

1.4 This test method is applicable to the approximate temperature range – 150 to 700°C. The temperature range may be extended depending on the instrumentation and calibration materials used.

1.5 The precision of measurement of this absolute method (better than ±40 nm/(m·K)) is significantly higher than that of comparative methods such as push rod dilatometry (for example, Test Methods D 696 and E 228) and thermomechanical analysis (for example, Test Method E 831) techniques. It is applicable to materials having low and either positive or negative coefficients of expansion (below 5 μm/(m·K)) and where only very limited lengths or thickness of other higher expansion coefficient materials are available.

1.6 Computer or electronic based instrumentation, techniques and data analysis systems equivalent to this test method can be used. Users of the test method are expressly advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user to determine the necessary equivalency prior to use.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appro-*

priate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:²

- D 696 Test Method for Coefficient of Linear Thermal Expansion of Plastics
- E 220 Test Method for Calibration of Thermocouples by Comparison Techniques
- E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer
- E 473 Terminology Relating to Thermal Analysis
- E 831 Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis
- E 1142 Terminology Relating to Thermophysical Properties

3. Terminology

3.1 Definitions:

3.1.1 The following terms are applicable to this document and are listed in Terminology E 473 and E 1142: coefficient of linear thermal expansion, thermodilatometry, thermomechanical analysis.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *thermal expansivity*, α_T , at temperature T , is calculated as follows from slope of length v temperature curve:

$$\alpha_T = \frac{1}{L_i} \lim_{T_2 \rightarrow T_1} \frac{L_2 - L_1}{T_2 - T_1} = \frac{1}{L_i} \frac{dL}{dT} \text{ with } T_1 < T_i < T_2 \quad (1)$$

and expressed as μm/m·K.

NOTE 1—Thermal expansivity is sometimes referred to as instantaneous coefficient of linear expansion.

3.2.2 *mean coefficient of linear thermal expansion*, α_m , the average change in length relative to the length of the specimen accompanying a change in temperature between temperatures T_1 and T_2 , expressed as follows:

$$\alpha_m = \frac{1}{L_0} \frac{L_2 - L_1}{T_2 - T_1} = \frac{1}{L_0} \frac{\Delta L}{\Delta T} \quad (2)$$

¹ This test method is under jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.05 on Thermophysical Properties.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

where:

α_m is obtained by dividing the linear thermal expansion ($\Delta L/L_0$) by the change of temperature (ΔT). It is normally expressed as $\mu\text{m}/\text{m}\cdot\text{K}$. Dimensions (L) are normally expressed in mm and wavelength (λ) in nm.

3.3 Symbols: Symbols:

3.3.1 α_m = mean coefficient of linear thermal expansion, see 3.2.1, $/K^1$.

3.3.2 α_T = expansivity at temperature T , see 3.2.2, $/K^1$.

3.3.3 L_0 = original length of specimen at temperature T_0 , mm.

3.3.4 L_1 = length at temperature T_1 , mm.

3.3.5 L_2 = length at temperature T_2 , mm.

3.3.6 ΔL = change in length of specimen between temperatures T_1 and T_2 , mm.

3.3.7 T_0 = temperature at which initial length is L_0 , K.

3.3.8 T_1, T_2 = two temperatures at which measurements are made, K.

3.3.9 ΔT = temperature difference between T_2 and T_1 , K.

3.3.10 N = number of fringes including fractional parts that are measured on changing temperature from T_1 to T_2 .

3.3.11 λ_v = wavelength of light used to produce fringes, nm.

3.3.12 n_r = index of refraction of gas at reference condition of temperature 288K and pressure of 100 kPa.

3.3.13 n = index of refraction of gas at temperature T and pressure, P .

3.3.14 n_1, n_2 = index of refractive of gas at temperature T_1 and T_2 , and pressure, P .

3.3.15 P = average pressure of gas during test, torr.

3.3.16 ΔL_s = change in length of reference specimen between T_1 and T_2 , mm.

4. Summary of Test Method

4.1 A specimen of known geometry can be given polished reflective ends or placed between two flat reflecting surfaces (mirrors). Typical configurations, as shown in Fig. 1, are a cylindrical tube or a rod with hemispherical or flat parallel ends or machined to provide a 3-point support. The mirrors consist

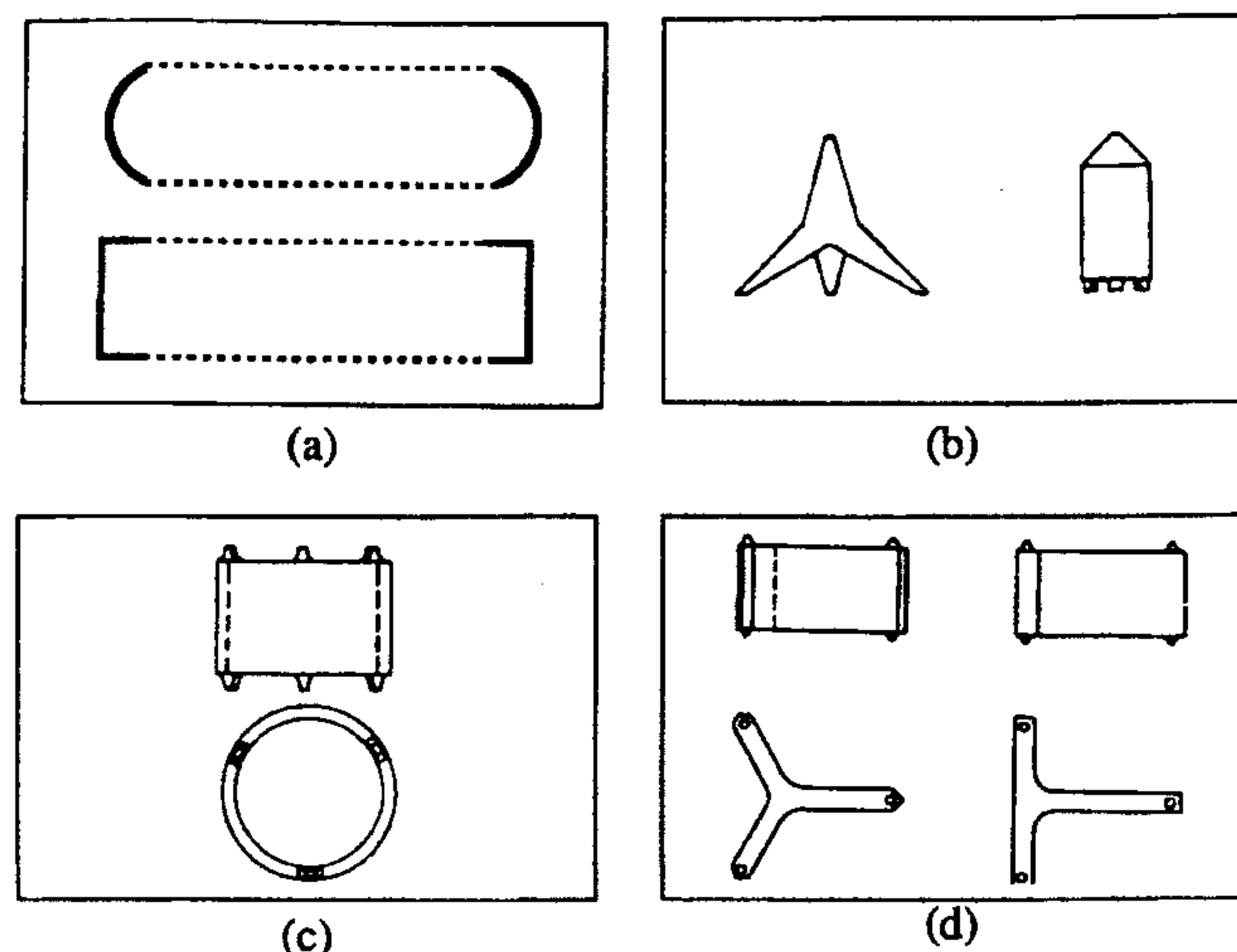


FIG. 1 Typical Specimen Configurations (a) Michelson Type, (b-d) Fizeau Type

of flat-uniform thickness pieces of silica or sapphire with the surfaces partially coated with gold or other high reflectance metal. Light, either parallel laser beam (Michelson, see Fig. 2 and Fig. 3) or from a point monochromatic source (Fizeau, see Fig. 4) illuminates each surface simultaneously to produce a fringe pattern. As the specimen is heated or cooled, expansion or contraction of the specimen causes a change in the fringe pattern due to the optical pathlength difference between the reflecting surfaces. This change is detected and converted into length change from which the expansion and expansion coefficient can be determined(1-5).

5. Significance and Use

5.1 Coefficients of linear expansion are required for design purposes and are used particularly to determine thermal stresses that can occur when a solid artifact composed of different materials may fail when it is subjected to a temperature excursion(s).

5.2 Many new composites are being produced that have very low thermal expansion coefficients for use in applications where very precise and critical alignment of components is necessary. Push rod dilatometry such as Test Methods D 696, E 228, and TMA methods such as Test Methods E 831 are not sufficiently precise for reliable measurements either on such material and systems, or on very short specimens of materials having higher coefficients.

5.3 The precision of the absolute method allows for its use to:

5.3.1 Measure very small changes in length;

5.3.2 Develop reference materials and transfer standards for calibration of other less precise techniques;

5.3.3 Measure and compare precisely the differences in coefficient of "matched" materials.

5.4 The precise measurement of thermal expansion involves two parameters; change of length and change of temperature. Since precise measurements of the first parameter can be made by this test method, it is essential that great attention is also paid to the second, in order to ensure that calculated expansion coefficients are based on the required temperature difference. Thus in order to ensure the necessary uniformity in temperature of the specimen, it is essential that the uniform temperature zone of the surrounding furnace or environmental chamber shall be made significantly longer than the combined length of specimen and mirrors.

5.5 This test method contains essential details of the design principles, specimen configurations, and procedures to provide precise values of thermal expansion. It is not practical in a method of this type to try to establish specific details of design, construction, and procedures to cover all contingencies that might present difficulties to a person not having the technical knowledge relating to the thermal measurements and general testing practice. Standardization of the method is not intended to restrict in any way further development of improved methodology.

5.6 The test method can be used for research, development, specification acceptance and quality control and assurance.

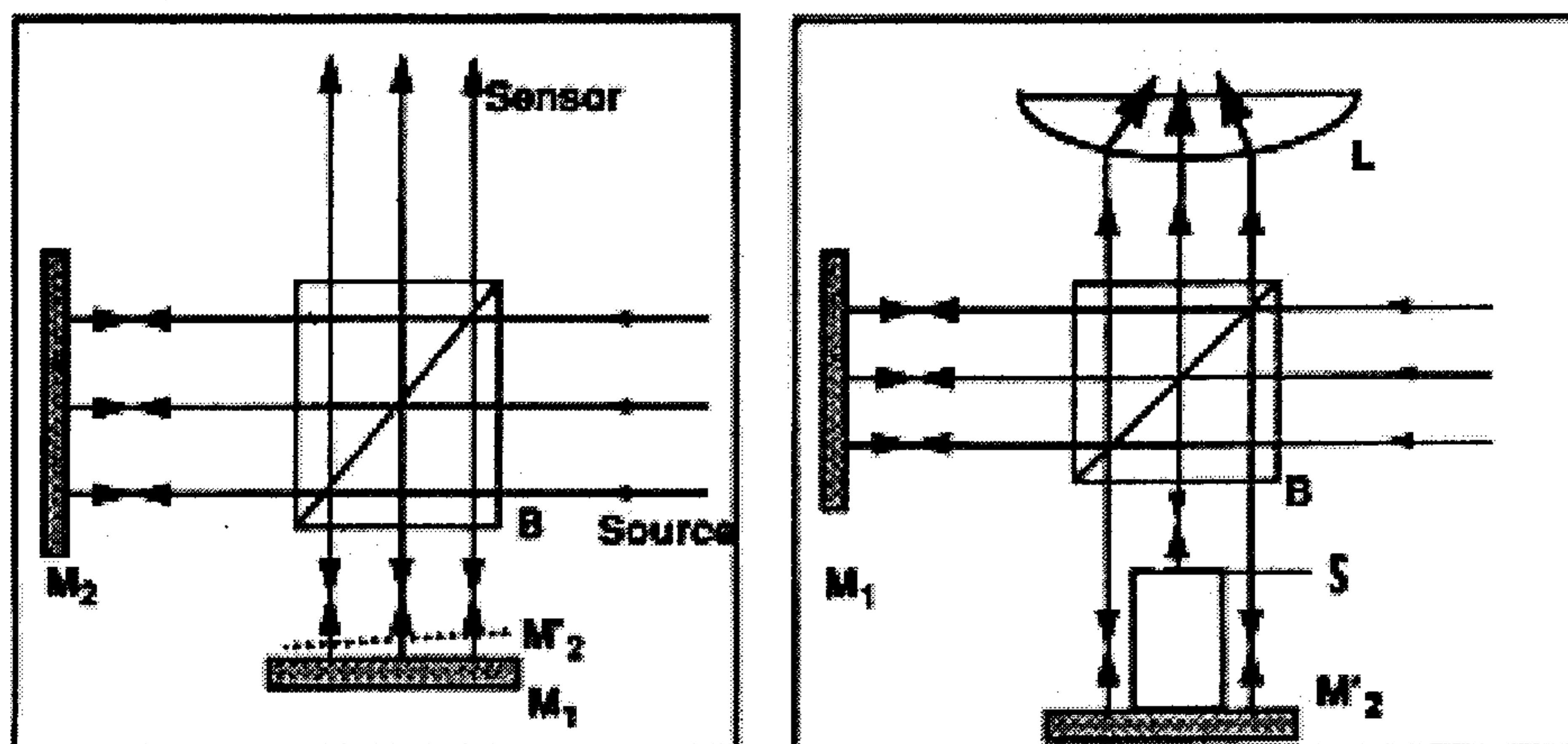


FIG. 2 (a) Principle of the Single Pass Michelson Interferometer, (b) Typical Single Pass System

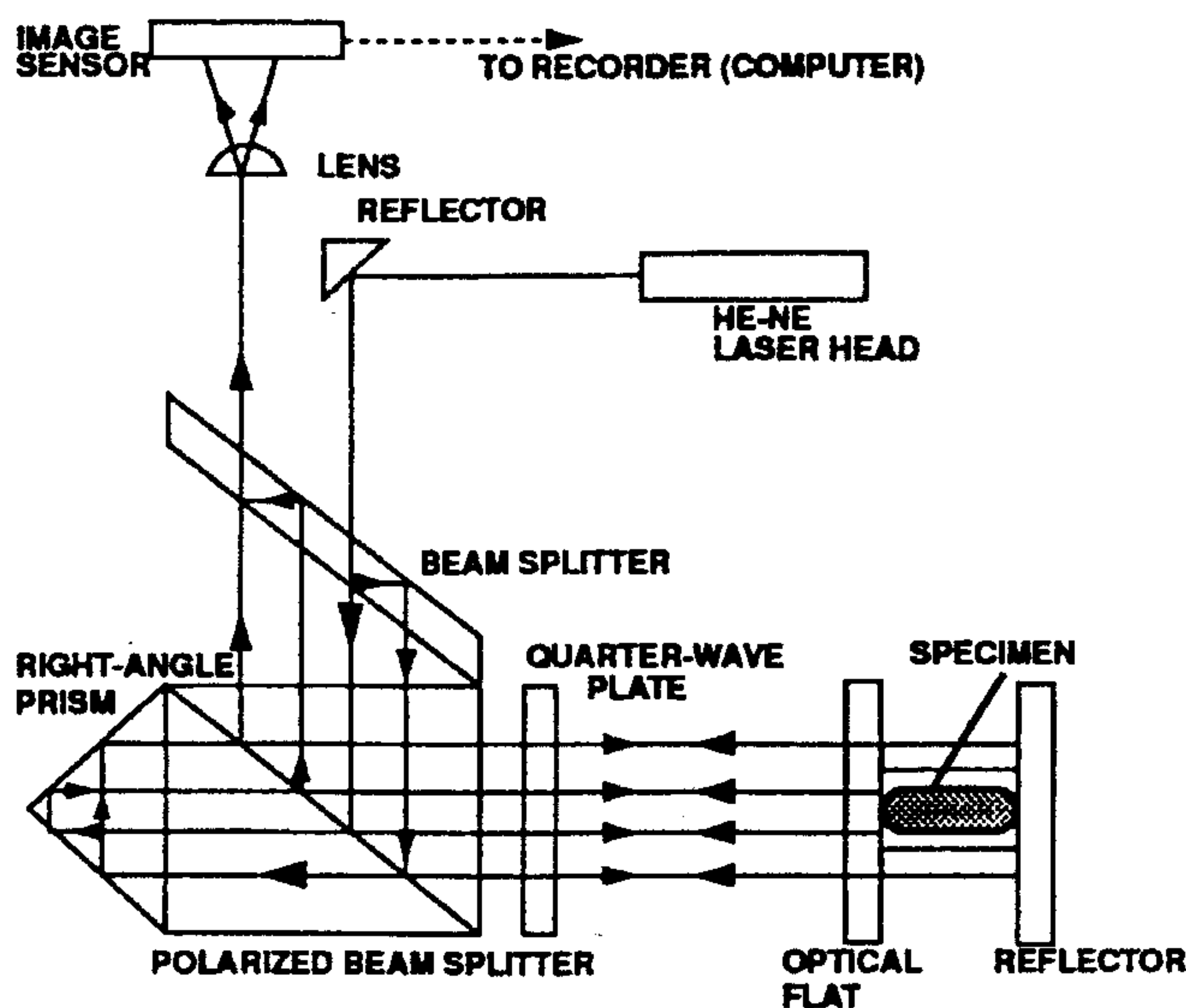


FIG. 3 Typical Double Pass Michelson Interferometer System

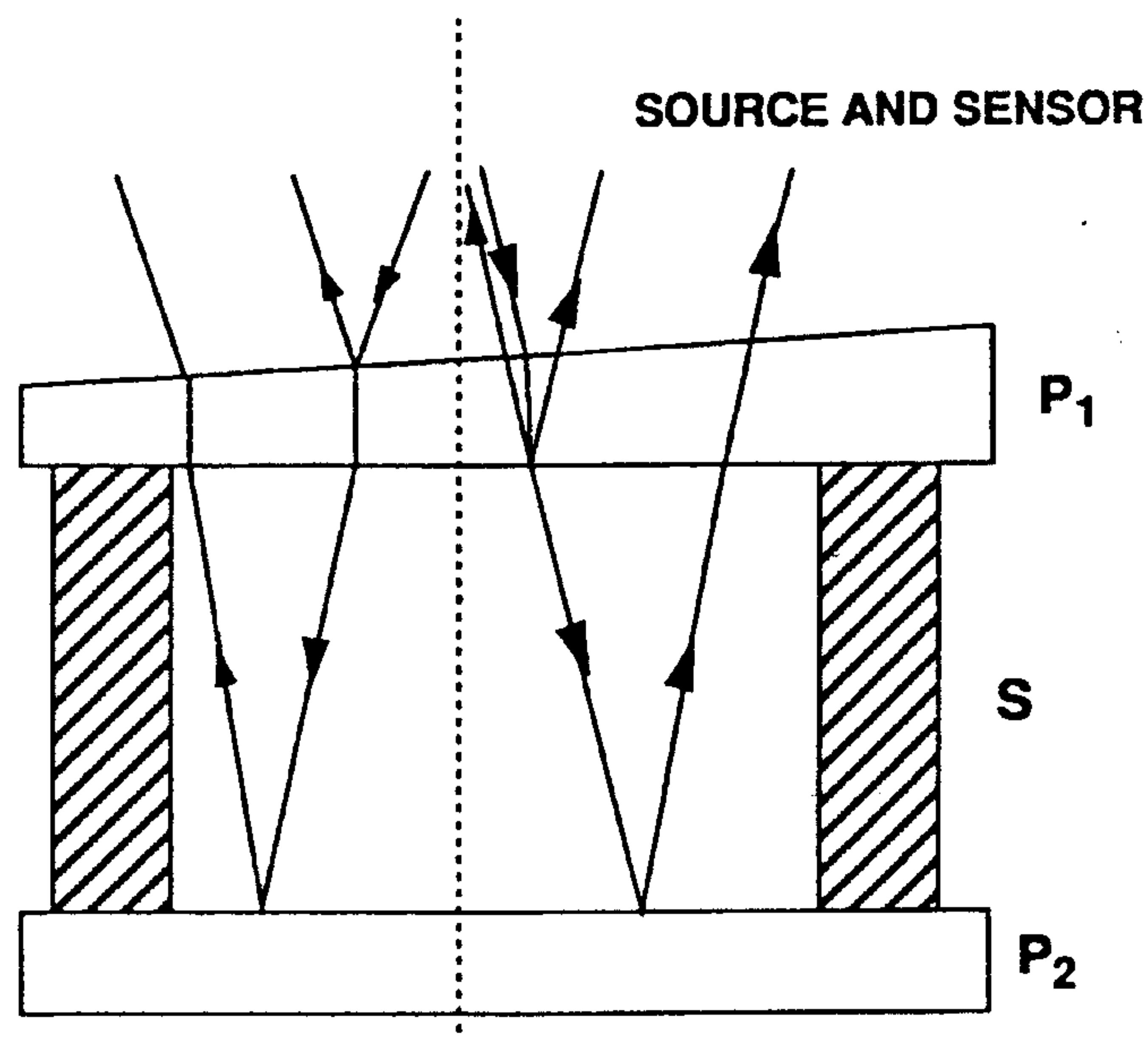


FIG. 4 Principle of the Fizeau Interferometer

6. Interferences

6.1 Measurements should normally be undertaken with the specimen in vacuum or in helium at a low gas pressure in order to off-set optical drifts resulting from instabilities of the refractive index of air or other gases at normal pressures. However, due to the reduced heat transfer coefficient from the surrounding environment, measurement in vacuum or low pressure can make actual specimen temperature measurement more difficult. Additional care and longer equilibrium time to ensure that the specimen is at a uniform temperature are necessary.

6.2 If vitreous silica flats are used, continuous heating to high temperatures may cause them to distort and become cloudy resulting in poor fringe definition.

7. Apparatus

7.1 Interferometer, Michelson Type:

7.1.1 The principle of the single pass absolute system is shown in Fig. 2a. A parallel light beam usually generated from a laser through a beam expander is split by a beam splitter B.

The resulting beams are reflected by mirrors M_1 and M_2 and recombined on B. If M'_2 is inclined slightly over the light-beam its mirror image M'_2 forms a small angle with M_1 producing fringes of equal thickness located on the virtual face M'_2 .

7.1.2 One example of a single contact type is shown in Fig. 2b. A prism or a polished very flat faced cylindrical specimen is placed on one mirror with one face also offered to the incident light. An interference pattern is generated and this is divided into two fields corresponding to each end of the specimen. The lens, L, projects the image of the fringes onto a plane where two detectors are placed one on the specimen and the other on the baseplate fields. As the specimen is heated or cooled, both the specimen and support change of lengths cause the surface S and M_2 to move relative to M_1 at different rates. The difference in the fringe count provides a measure of the net absolute expansion.

7.1.3 The principle of the double pass system is essentially similar to the single pass with three important distinctions. The specimen can be a relatively simple cylinder with hemispherical or flat ends and requiring less precise machining, the interfering beams are reflected twice from each face to the specimen thus giving twice the sensitivity of the single pass, and no reference arm is required. One example of the double pass form is shown in Fig. 3.

7.1.4 It is common practice to use polarized laser light and quarter wave plates to generate circularly polarized light. In this way detectors combined with appropriate analyzers generate signals either with information on fringe number, fraction and motion sense for each beam or linear array data of light intensity, which indicate the profile of the instantaneous whole fringe pattern. The array data provides complete information (position of fringe and distance between fringes) to determine the absolute length change of the specimen depending upon the system. These signals are normally processed electronically.

7.2 Fizeau Type:

7.2.1 This type is available in both absolute and comparative versions.

7.2.2 The principle of the absolute method is illustrated in Fig. 4. The specimen is retained between two parallel plates and illuminated by the point source. Expansion or contraction of the specimen causes spatial variation between the plates and radial motion of the circular fringe pattern.

7.2.3 The difference in the fringe counts yields the net absolute expansion of the specimen.

7.2.4 In practice, P_1 is wedge shaped (less than 30 min of arc) such that light reflected by the upper face is diverted from the viewing field, while the lower face of P_2 is made to absorb the incident light, depending upon the total separation of the flats.

7.2.5 For use in the comparative mode, two forms are available. These are described in detailed in Annex A1.

7.3 Furnace/Cryostat:

7.3.1 Fig. 5 and Fig. 6 illustrate the construction of a typical vertical type of furnace and cryostat that are suitable for use in undertaking these measurements. For the double pass Michelson system, horizontal forms of furnace and cryostat can be used.

7.4 Temperature Measurement System:

7.4.1 The temperature measurement system shall consist of a calibrated sensor or sensors together with manual, electronic or equivalent read-out such that the indicated temperature can be determined better than $\pm 0.5^\circ\text{C}$.

7.4.1.1 Since this method is used over a broad temperature range, different types of sensors may have to be used to cover the complete range. The common sensor(s) is a fine gage (32 AWG or smaller wire) or thin foil thermocouples calibrated in accordance with Test Method E 220.

7.4.1.2 Types E and T are recommended for the temperature range -190 to 350°C and Types K and S and Nicrosil for the temperature range from 0 to 800°C . If Type K is used continuously, regular checking of the calibration should be undertaken to ensure that contamination or phase change phenomena due to alloy component migration from the junction has not taken place during testing.

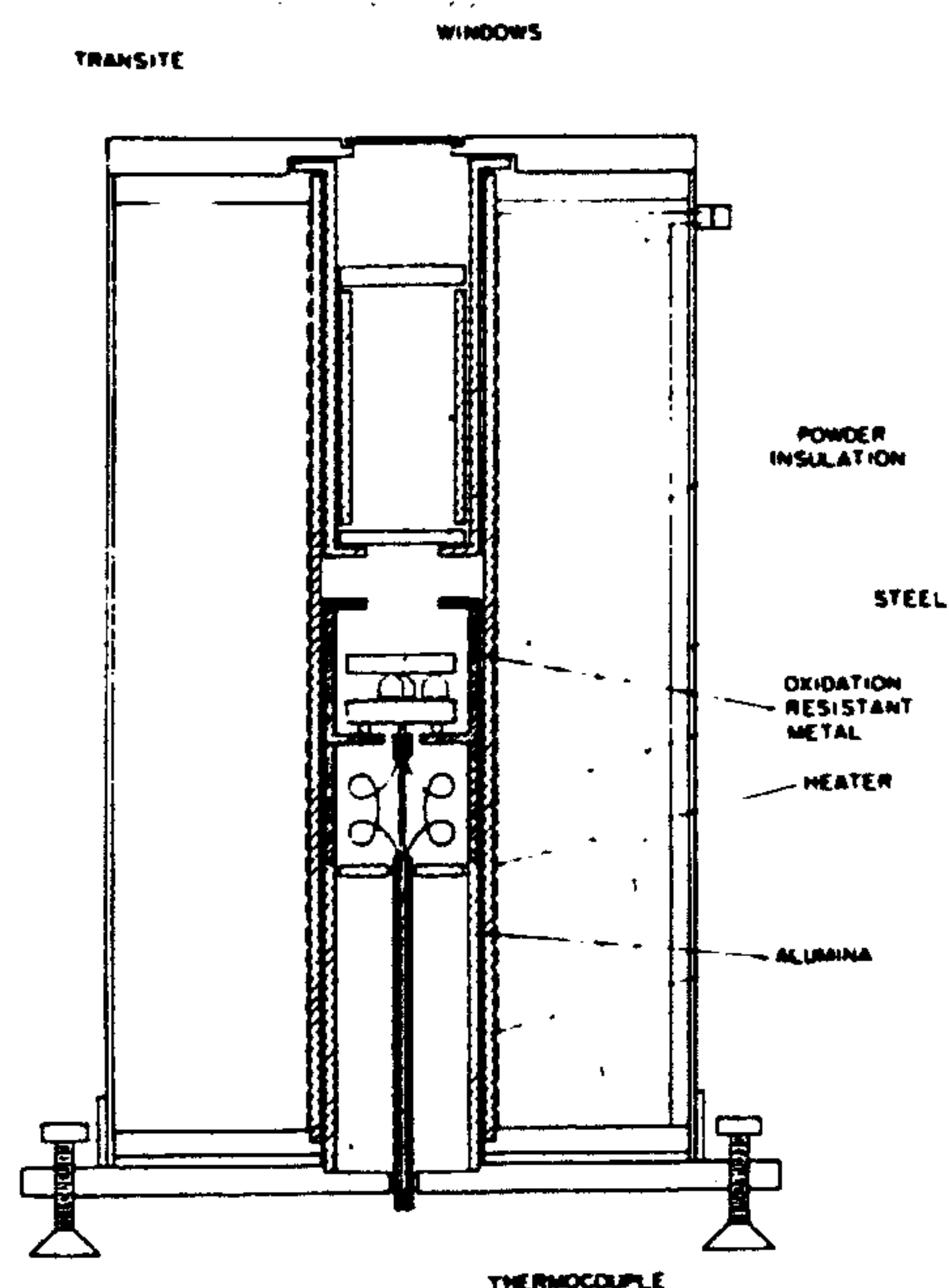


FIG. 5 Typical Furnace

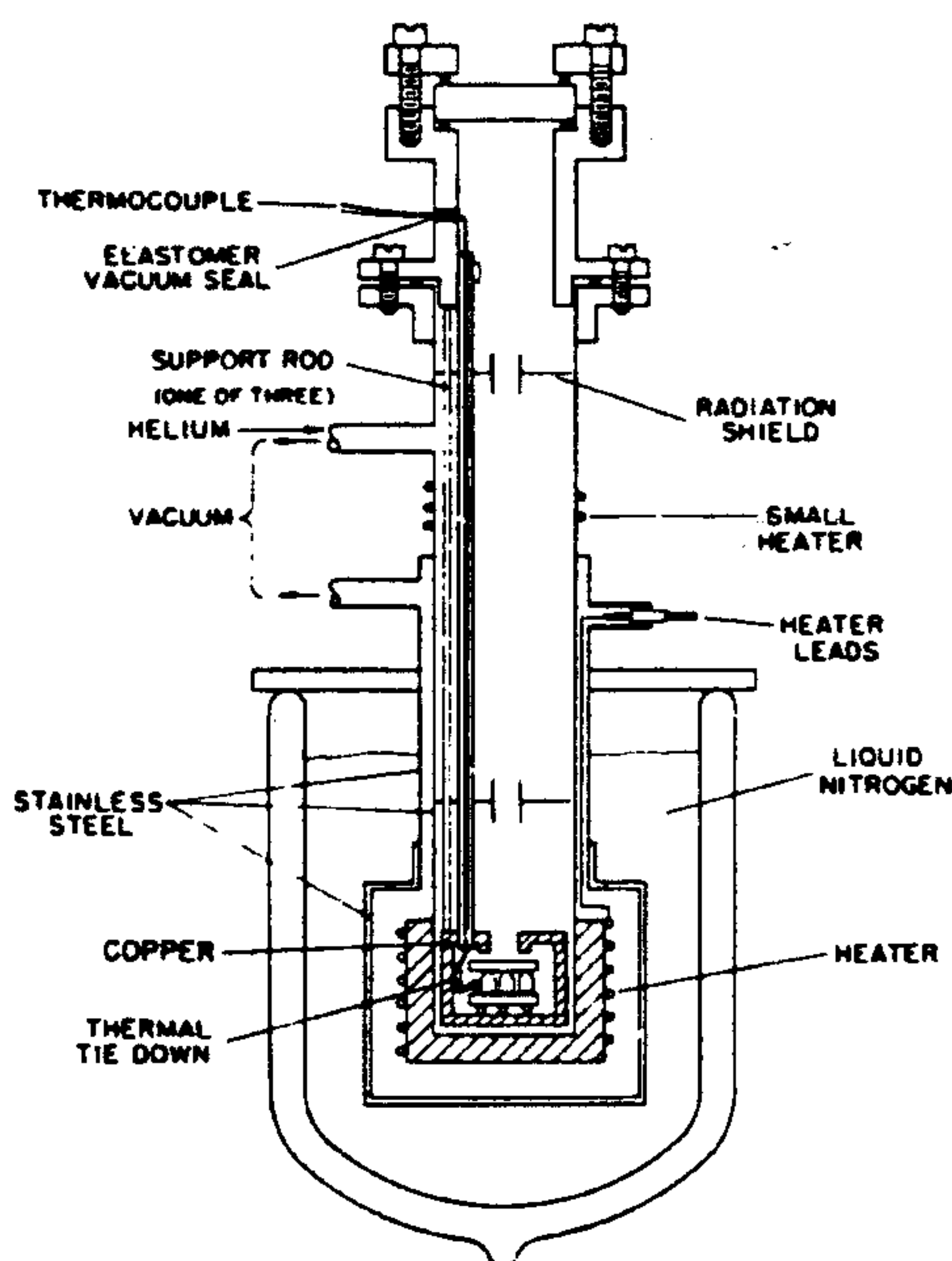


FIG. 6 Typical Low-Temperature Cryostat

7.4.1.3 In all cases where thermocouples are used they shall be referenced to 0°C by means of an ice water bath or equivalent electronic reference system, insulated from the effects of temperature variations in the immediate surrounding ambient.

7.4.1.4 For temperatures below -190°C a calibrated carbon or germanium resistance thermometer is used.

7.5 A measurement instrument such as an index micrometer or calipers capable of reading to 0.01 mm in order to determine the initial and final lengths of the test specimen (and other

relevant components where required, see Section 8.1) and adjusting the specimen length originally to obtain fringes.

8. Test Specimen

8.1 The specimen shall be selected from a sample in accordance with the sampling requirements of the appropriate materials standard. If possible, it may be fabricated in one of the forms shown. For the Michelson interferometer, the form with rounded ends shown in Fig. 1(a) gives point contact. For flat panels, mirrors may be attached as described in (7). Configurations 1b–1d are those based on 3-point support that is most appropriate for the Fizeau interferometer. The legs must be ground flat such that they are all of the same length as measured by the micrometer.

NOTE 2—Conditioning of specimens is often necessary before reproducible expansion data can be obtained. For example, heat treatments are frequently necessary to eliminate certain effects (strain, moisture, and the like), which may introduce length changes not associated with thermal expansion.

8.1.1 Where possible, the specimen length should be at least 5 mm, but short enough to allow for temperature uniformity of the specimen. For Michelson interferometry, the sample length is limited by the coherence length of the light source. Some light sources have considerably larger coherence length, for

example, The optimal length is between 10 and 20 mm. For the double pass type where a cylindrical specimen is used the radius of curvature of the hemispherical ends should be 3 mm or less and the diameter uniform to ±1 m rad.

8.1.2 Where only shorter specimens are available such as sheet and foil materials, the double pass Michelson interferometer can be used by including two equal thickness quartz glass pieces each with a rounded end and optically flat surface (see 7.1). The pieces should be of an appropriate thickness such that the total thickness of a sandwich of the test flat specimen and quartz pieces is within the optimal range.

8.2 Where only thinner or shorter specimens are available, when testing materials that exhibit different properties in different directions, special care must be taken when preparing the pin or pyramidal type specimens to ensure that all three have the same angle between their axes and the principal axis of anisotropy.

9. Verification

9.1 The Michelson and Fizeau interferometers determine dimensional length change absolutely. However, it is essential that the system be verified by undertaking measurement on known standard(s) for which the thermal expansion has been verified. Table 1 and Table 2 contains details of the linear

TABLE 1 (a) Thermal Expansion of Various Reference Materials

Temperature° K	SRM 731 (Borosilicate Glass) ^A		SRM 738 (Stainless Steel) ^A		SRM 739 (Fused Silica) ^A	
	Expansion 10 ⁶ Δ L/L ₂₉₃	10 ⁶ Expansivity	Expansion 10 ⁶ Δ L/L ₂₉₃	10 ⁶ Expansivity	Expansion 10 ⁶ Δ L/L ₂₉₃	10 ⁶ Expansivity
80	-819	-1	-0.07
100	-771	2.64	-13	-0.53
120	-714	3.07	-22.5	-0.38
140	-649	3.43	-28.5	-0.24
160	-578	3.72	-32	-0.10
180	-501	3.97	-32.5	0.02
200	-419	4.17	-31	0.13
220	-334	4.34	-27.5	0.23
240	-246	4.48	-22	0.32
260	-155	4.60	-14	0.39
280	-62	4.71	-6	0.45
293	0	4.78	0	9.76	0	0.48
300	34	4.82	69	9.81
320	131	4.91	13.5	0.53
340	230	4.99	466	10.04	24.5	0.56
380	432	5.11	872	10.28	47.5	0.60
420	638	5.19	1288	10.52	72	0.62
460	847	5.23	1714	10.76	97	0.63
500	1057	5.26	2149	11.00	122	0.63
540	1267	5.27	2593	11.23
560	1372	5.27	159	0.61
580	1478	5.27	3048	11.47
600	1583	5.27	183	0.59
620	1689	5.28	3511	11.71
640	1794	5.29	206	0.56
660	1900	...	3984	11.95
680	2007	228	0.54
700	4467	12.19
720	249	0.51
740	4959	12.42
760	269	0.49
780	5461	12.66
800	228	0.47
840	307	0.44
880	324	0.42
920	340	0.40
960	356	0.38
1000	371	0.37

^AAvailable from National Institute of Standards and Technology, Gaithersburg, MD. Values in table from relevant certificate for the reference material.

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TABLE 2 (b) Reference Kieselglas K001^A

Temperature ^a C	Expansion		Expansivity		10 ⁶ α	
	10 ⁶ ΔL/L ₀		10 ⁶ α (t)		K ⁻¹	
-170	-2.16	±0.67	-0.565	±0.019	0.013	±0.004
-160	-7.35	±0.55	-0.475	±0.016	0.046	±0.003
-140	-15.15	±0.44	-0.300	±0.011	0.108	±0.003
-120	-19.80	±0.43	-0.160	±0.007	0.165	±0.004
-100	-21.67	±0.43	-0.030	±0.005	0.217	±0.004
-80	-21.09	±0.43	0.085	±0.004	0.264	±0.006
-60	-18.36	±0.41	0.185	±0.004	0.306	±0.007
-40	-13.76	±0.38	0.272	±0.004	0.344	±0.010
-20	-7.57	±0.36	0.346	±0.005	0.378	±0.019
-10	-3.94	±0.34	0.379	±0.005	0.394	±0.038
0	0.00	±0.34	0.409	±0.005	***	***
10	4.23	±0.33	0.436	±0.005	0.423	±0.038
20	8.71	±0.33	0.46	±0.004	0.436	±0.019
40	18.38	±0.35	0.504	±0.004	0.459	±0.010
60	28.81	±0.37	0.538	±0.004	0.480	±0.007
80	39.86	±0.39	0.565	±0.003	0.498	±0.006
100	51.37	±0.41	0.585	±0.003	0.514	±0.005
120	63.22	±0.43	0.599	±0.003	0.527	±0.004
140	75.31	±0.44	0.608	±0.003	0.538	±0.004
160	87.52	±0.44	0.612	±0.003	0.547	±0.003
180	99.78	±0.45	0.613	±0.003	0.554	±0.003
200	112.01	±0.45	0.610	±0.004	0.560	±0.003
220	124.16	±0.45	0.605	±0.004	0.564	±0.002
240	136.18	±0.46	0.567	±0.004	0.567	±0.002
260	148.03	±0.47	0.588	±0.004	0.569	±0.002
280	159.68	±0.48	0.577	±0.004	0.570	±0.002
300	171.10	±0.50	0.565	±0.004	0.570	±0.002

^AFused Silica—Available from Physikalisch Technische Bundesanstalt, Bundesallee 400, 3300 Braunschweig, Germany. Values in table from relevant certificate for reference material.

expansion of several reference materials available from two national standards organizations.

9.2 The temperature uniformity over the specimen length and the heating uniformity of the heating rate also should be established with a specimen instrumented with the appropriate temperature sensor(s).

10. Procedure

10.1 Set-Up:

10.1.1 Michelson Interferometer:

10.1.1.1 Measure the initial length of the specimen after carefully cleaning with a solvent.

10.1.1.2 Switch on the light source.

10.1.1.3 Insert the specimen between the two flats (mirrors), or attach mirrors, mount in the furnace or cryostat. The mirrors must remain parallel in order to obtain a fringe pattern. For the double pass system, the cylindrical specimen is aligned parallel to the axis of the expansion measurement. This is usually accomplished by means of an appropriate combined parallel spring mechanism having minimal or no frictional force and with the specimen placed in a special jig. Under these circumstances and with the specimen having point contacts on the mirror surfaces, the parallel spring mechanism will ensure that the mirrors remain parallel during the whole test (see Note 3).

NOTE 3—Experience has shown that the diameter of the specimen should be uniform to ± 1 m rad and the radius of curvature of the hemispherical ends should be 3 mm or less.

10.1.1.4 Establish a zero point on one fringe of the pattern.

10.1.2 Fizeau Interferometer:

10.1.2.1 Mount the clean specimen between the optical flats and place in the furnace or cryostat. (Cleaning is critical).

10.1.2.2 Switch on the light source.

10.1.2.3 Observe the fringe pattern. If a strong wide fringe pattern is not obtained, further adjustment of one or more of the support legs is required. Thus the length of the legs requires further machining until an adequate pattern is obtained (Note 4).

NOTE 4—When using visual or photographic methods of viewing, about four fringes per centimeter is the optimum number to have in the field of view. A larger number of fringes may be more satisfactory when using photoelectric techniques. If possible, the specimen should be placed between the flats so that one of the three points of support bears most of the weight of the top flat. After insertion in the furnace or cryostat, a gentle tap may be necessary to restore the fringe pattern.

10.1.2.4 The position directly over the support that bears most of the weight of the top flat is normally chosen as the reference point of the fringe system for subsequent measurements. This point is established by prior chemical etching of the surface of one flat.

10.2 Attach the sensor(s) or thermocouple(s) to any appropriate part of the specimen/mirror combinations such that it does not disturb the interferometer or subject the specimen to any mechanical strain. If this is not possible, place the junction, as close to the specimen as can be arranged. An acceptable position is directly under or close to a flat within 0.5 mm of the surface.

10.2.1 Appendix X1 contains additional information relating to measurement of temperature.

10.3 Evacuate the system, and where appropriate backfill with helium gas to 10 torr maximum. Other gases at appropriate pressures may be used, but their use should be referenced in the report and allowance shall be made for the effects of pressure and temperature on the index of refraction of the gas. A total pressure of < 100 mTORR is recommended

10.4 Allow the system to attain equilibrium, measure the temperature and note the position of a particular fringe (Michelson) or its relation to the reference point (Fizeau) with an electronic or manual detector.

10.5 Measurement

10.5.1 The most precise measurement is accomplished by heating the specimen successively to a number of regular incremental constant temperatures, allowing the system to equilibrate and counting the number of fringes including fractional parts passing the origin of the detector or the reference point.

10.5.2 Alternatively, the specimen should be heated at a constant temperature rate (less than 3°C/min) and a continuous record of the number of fringes including fractional parts, and of the temperature, should be obtained (Note 5).

10.5.3 Measurements should be undertaken to establish that the specimen has not undergone any permanent change in length due to the heating or colling received during the test.

NOTE 5—In material control testing, the heating or cooling rates should be highly repeatable. It is recommended that an automatic fringe counting system should be used, which makes a sampling of the whole linear image of the fringe pattern using a photoelectric transducer array (for example, CCD) and makes image processing by computer to detect the position and distance of the fringes in order to calculate the absolute length change as

a continuous value using the wavelength of the light.

11. Calculation

11.1 Calculate the linear thermal expansion for each temperature interval as follows:

11.1.1 Fizeau and Single Pass Michelson:

$$\frac{\Delta L}{L_0} = \frac{N\lambda_v}{2L_0} \quad (3)$$

11.1.2 Double Pass Michelson:

$$\frac{\Delta L}{L_0} = \frac{N\lambda_v}{4L_0} \quad (4)$$

11.1.3 Where measurements are carried out at helium or other gas pressures above 1.33 kPa allowance should be made for the effects of pressure and temperature on the index of refractions of the gas. As an example, for the Fizeau types:

$$\frac{\Delta L}{L_0} = \frac{N\lambda_v}{2L_0 n_2} + \frac{n_1 - n_2}{n_2} \quad (5)$$

and

$$n - 1 = (n_r - 1)^{(288P/760T)} \quad (6)$$

At the 288 k and 100 kPa reference conditions, for air $n_r = 1.0002771$ (yellow line helium) or 1.0002779 (green line of mercury). For helium $n_r = 1.000036$.

For helium at a pressure of 1.33 kPa or less, the overall effect is less than ± 6 nm and may be neglected.

NOTE 6—This equation should not be used for calculation of the index of refraction of air below 293 k.

11.1.4 Calculate the values of α_m or α by using the measured values of 10.1.1 or 10.1.2 together with the appropriate temperature difference ($T_1 - T_2$).

11.1.5 In calculation of the relevant quantities, use all available decimal places for each parameter through to the final result and report data to two significant figures (Ref. 12.1.5 and 12.1.6).

12. Report

12.1 The report shall include the following:

12.1.1 Description of material, manufacturer, chemical composition, thermal, and mechanical history;

12.1.2 Method of preparation of test specimen, axis orientation if material is anisotropic, together with details of any subsequent thermal, mechanical, moisture, or other conditioning;

12.1.3 Form and dimensions of test specimen, including initial length L_0 , and reference temperature T_0 ;

12.1.4 Brief description of apparatus, including displacement and temperature-measuring systems, estimate of precision, heating and cooling rates, temperature controls, and atmosphere;

12.1.5 Tabulation of data showing linear thermal expansion test temperatures and pressures, and values for mean coefficient of linear thermal expansion for selected temperature intervals;

12.1.6 The following curves may be plotted as required:

$$\Delta L/L_0 \text{ versus } T; \alpha_m \text{ versus } T; \alpha_T \text{ versus } T \quad (7)$$

where α_m is computed from a common reference temperature to T ;

12.1.7 Complete description of any unusual behavior of the specimen, such as permanent change in specimen length at reference temperature following test, excessive oxidation, scaling, discoloration, deformation, cracking, spalling, and the like, which may be of value in interpreting test results, and;

12.1.8 Any additional information required by a particular material specification.

13. Precision and Bias

13.1 An interlaboratory comparison of Michelson double pass and Fizeau type absolute interferometer has been made between -150 and 150°C at 50°C intervals on specimens of two low thermal expansion glasses using a highly sensitive double pass system at the National Research Laboratory of Metrology in Japan as the reference system (6). The study included replication of specimen, effects of heating and cooling rates and direction of temperature increase and decrease.

13.2 The results of the study indicated that a precision of measurement of better than $\pm 4 \times 10^{-8} \text{ K}^{-1}$ can be obtained with either system with no discernible bias.

13.3 In the development of thermal expansion reference materials SRM 731, 738, and 739 by the Standard Reference Material Program of the National Institute of Standards and Technology, using Fizeau interferometry, the results of multi-specimen, multi-run experiments indicated that the measurement precision was $\pm 3 \times 10^{-8} \text{ K}^{-1}$.

14. Keywords

14.1 coefficient of linear thermal expansion; contraction; expansion; Fizeau interferometer; interferometry; Michelson interferometer

A1. COMPARATIVE TYPE FIZEAU INTERFEROMETERS

A1.1 Two types of the comparative Fizeau interferometers are also used to measure thermal expansion. The expansion is measured relative to that of a reference material such as vitreous silica.

A1.2 The interferometers are the Abbe-Pulfrich and Priest (Dental) shown in Fig. A1.1a and Fig. A1.1b respectively.

A1.2.1 The Abbe-Pulfrich interferometer requires specimens of the pin or pyramidal configuration and the Priest uses only the ring or pin forms shown in Fig. 1.

A1.2.2 The former requires a reference point and the latter two reference lines, which are established by chemical etching of an appropriate glass surface. The reference point is established as the geometric center of the system. In the case of the Priest interferometer, the points of intersection of two parallel reference lines and perpendicular connecting line placed on the lower surface of the top flat (or on the top surface of the lower flat) may be used.

A1.2.2.1 When the temperature of the Abbe-Pulfrich interferometer is changed, the number of fringes passing the reference point is a measure of the change in length of the specimen.

A1.2.2.2 For the Priest interferometer, the change in length is indicated by the change in the number of fringes between the reference lines.

A1.3 Prior calibration of these interferometers using a reference specimen is required in order to obtain the change in length of a test specimen. The correction for the reference material is determined by using one of the calibration materials

listed in Table 1 and heating and cooling it over the desired temperature range in accordance with the criteria outlined in Section 9. The difference between the calibration and measured values represents the linear expansion of the reference material.

A1.4 Since the calibration has been established, measurements are made on a test specimen in accordance with the procedures in Section 9.

A1.4.1 For the Abbe-Pulfrich interferometer the linear expansion for each reading is calculated as follows:

$$\frac{\Delta L}{L_0} = \frac{\Delta L_s}{L_0} = \frac{N\lambda_v}{2L_0n_1} \quad (A1.1)$$

A1.4.2 For the Priest interferometer, it is calculated as follows:

$$\frac{\Delta L}{L_0} = \frac{\Delta L_s}{L_0} + \frac{D\lambda_v}{2dL_0n_1(N_2 - N_1)} \quad (A1.2)$$

In this particular case:

- D = the perpendicular distance between the specimen bearing point and the line joining the reference lines (Fig. A1.1b), mm,
- d = the perpendicular distance between the reference lines, mm, and
- N_1 and N_2 = the number of fringes, including fractional parts, between the reference lines at temperatures T_1 and T_2 .

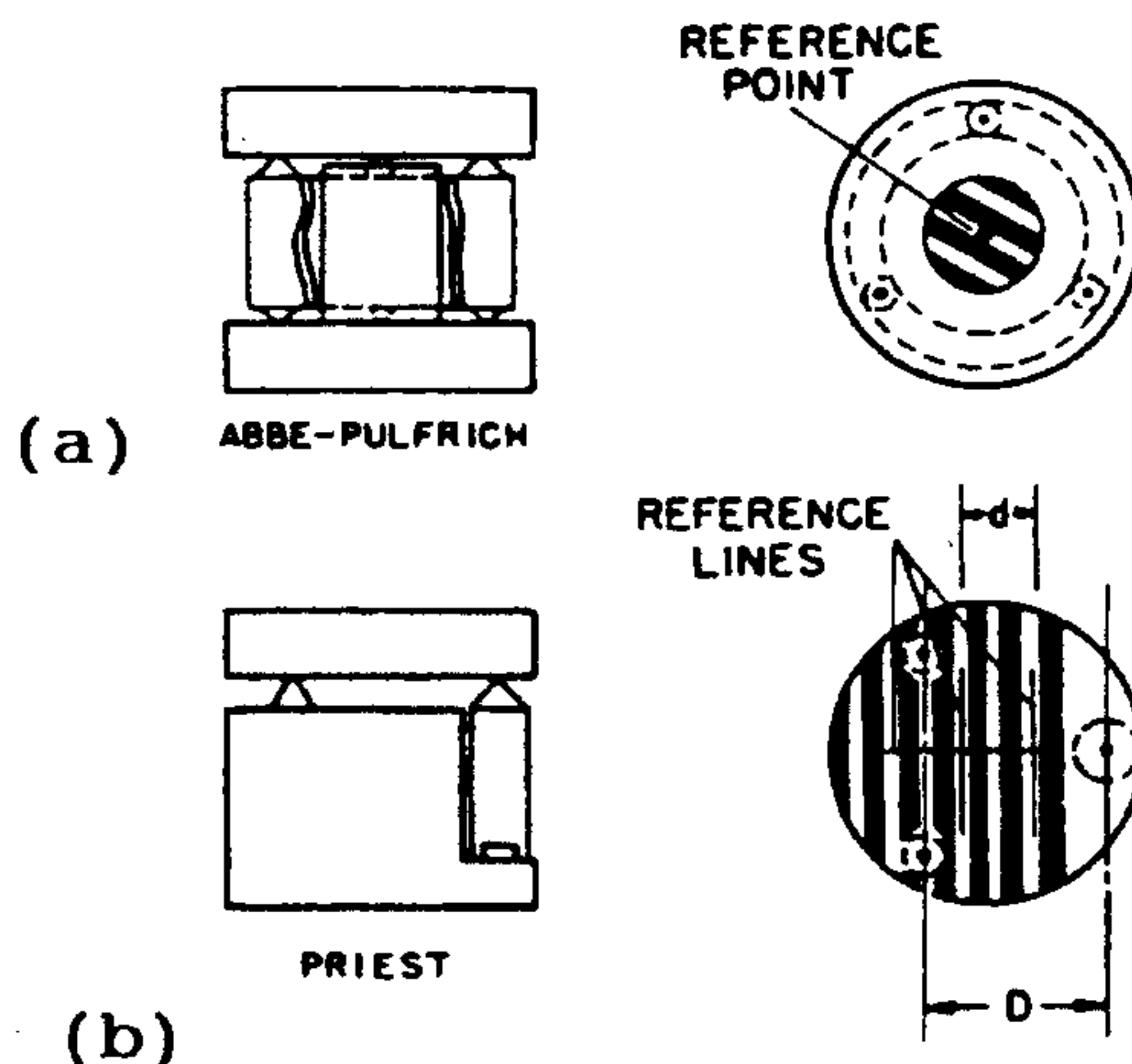


FIG. A1.1 Two Comparative (Relative) Type Fizeau Interferometers

APPENDIX

(Nonmandatory Information)

X1. TEMPERATURE DETERMINATION

X1.1 Special care must be taken when the sensor(s) is not in direct contact with the specimen, for even when it is in good thermal contact, the measured temperature can still be in error. This is caused by heat being conducted away from or toward the specimen or the thermocouple junction, or both, by the thermocouple wires. This heat flow can be minimized by reducing the temperature gradient along the thermocouple wires near the junction. For example, some of the wire is looped once or twice within an area that has a temperature very close to that of the specimen. The temperature gradient is thereby shifted to an area between an intermediate point on the wire and the outside of the furnace.

X1.2 When the junction is not in direct contact with the specimen, the temperature measurements may also be in error when heating or cooling. The magnitude of this sensor lead or lag usually is not great but will be dependent on its distance from the specimen, the specimen size, the emittance and

thermal diffusivity of the material and on the heating or cooling rate.

X1.3 The sensor lead and lag can, however, be determined in the following manner:

X1.3.1 Where the specimen to be measured is known or shown to be reversible in its thermal expansion behavior, heat the interferometer at a constant rate over the temperature range of interest and then cool through this region at the same rate. A plot of the expansion versus apparent temperature will show a temperature displacement between the heating and cooling data. One half of this displacement parallel with the temperature axis is the lead and lag correction. Subtract this correction from apparent temperature on heating and add on cooling. If the specimen is not reversible in thermal expansion behavior, another material of similar emittance and thermal diffusivity and known to be reversible may be substituted to determine the approximate lead and lag.

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