



Designation: C 1211 – 98a

Standard Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures¹

This standard is issued under the fixed designation C 1211; 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 determination of the flexural strength of advanced ceramics at elevated temperatures.² Four-point- $\frac{1}{4}$ point and three-point loadings with prescribed spans are the standard. Rectangular specimens of prescribed cross-section are used with specified features in prescribed specimen-fixture combinations.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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 appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

C 1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature³

C 1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics³

C 1341 Test Method for Flexural Properties of Continuous Fiber Reinforced Advanced Ceramic Composites³

C 1368 Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Ambient Temperature³

E 4 Practices for Force Verification of Testing Machines⁴

E 220 Method for Calibration of Thermocouples by Comparison Techniques⁵

E 230 Temperature Electromotive Force (EMF) Tables for Standardized Thermocouples⁵

2.2 Military Standard:

MIL-STD 1942(A) Flexural Strength of High Performance Ceramics at Ambient Temperature⁶

3. Terminology

3.1 Definitions:

3.1.1 *flexural strength*—a measure of the ultimate strength of a specified beam in bending.

3.1.2 *four-point- $\frac{1}{4}$ point flexure*—a configuration of flexural strength testing in which a specimen is symmetrically loaded at two locations that are situated at one-quarter of the overall span, away from the outer two support bearings (see Fig. 1).

3.1.3 *inert flexural strength, n* —a measure of the strength of a specified beam specimen in bending as determined in an appropriate inert condition whereby no slow crack growth occurs.

3.1.4 *slow crack growth (SCG), n* —Subcritical crack growth (extension) which may result from, but is not restricted to, such mechanisms as environmentally-assisted stress corrosion or diffusive crack growth.

3.1.5 *three-point flexure*—a configuration of flexural strength testing in which a specimen is loaded at a position midway between two support bearings (see Fig. 1).

4. Significance and Use

4.1 This test method may be used for material development, quality control, characterization, and design data generation purposes. This test method is intended to be used with ceramics whose flexural strength is ~ 50 MPa (~ 7 ksi) or greater.

4.2 The flexure stress is computed based on simple beam theory, with assumptions that the material is isotropic and homogeneous, the moduli of elasticity in tension and compression are identical, and the material is linearly elastic. The average grain size should be no greater than $\frac{1}{50}$ of the beam thickness. The homogeneity and isotropy assumptions in the

¹ This test method is under the jurisdiction of ASTM Committee C-28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.01 on Properties and Performance.

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² Elevated temperatures typically denote, but are not restricted to 200 to 1600°C.

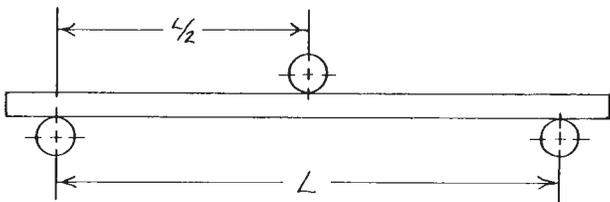
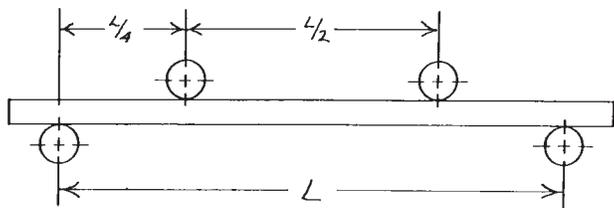
³ *Annual Book of ASTM Standards*, Vol 15.01.

⁴ *Annual Book of ASTM Standards*, Vol 03.01.

⁵ *Annual Book of ASTM Standards*, Vol 14.03.

⁶ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094. This document is a 1990 update of the original MIL-STD 1942(MR), dated November 1983.

*A Summary of Changes section appears at the end of this standard.



NOTE 1—Configuration:

- A: L = 20 mm
- B: L = 40 mm
- C: L = 80 mm

FIG. 1 Four-Point-1/4 Point and Three-Point Fixture Configurations

test method rule out the use of it for continuous fiber-reinforced composites for which Test Method C 1341 is more appropriate.

4.3 The flexural strength of a group of test specimens is influenced by several parameters associated with the test procedure. Such factors include the testing rate, test environment, specimen size, specimen preparation, and test fixtures. Specimen and fixture sizes were chosen to provide a balance between the practical configurations and resulting errors as discussed in MIL-STD 1942(A), Test Method C 1161, and Refs (1–3).⁷ Specific fixture and specimen configurations were designated in order to permit the ready comparison of data without the need for Weibull size scaling.

4.4 The flexural strength of a ceramic material is dependent on both its inherent resistance to fracture and the size and severity of flaws that are present. Fractographic analysis of fracture surfaces, although beyond the scope of this test method, is highly recommended for all purposes, especially for design data. See Practice C 1322.

4.5 Flexure strength at elevated temperature may be strongly dependent on testing rate, a consequence of creep, stress corrosion, or slow crack growth. This test method measures the flexural strength at high loading rates in order to minimize these effects.

5. Interferences

5.1 Time-dependent phenomena, such as stress corrosion and slow crack growth, can interfere with determination of the flexural strength at room and elevated temperatures. Creep phenomena also become significant at elevated temperatures. Creep deformation can cause stress relaxation in a flexure

TABLE 1 Fixture Spans

Configuration	Support Span (L), mm	Loading Span, mm
A	20	10
B	40	20
C	80	40

specimen during a strength test, thereby causing the elastic formulation that is used to compute the strength to be in error.

5.2 Surface preparation of the test specimens can introduce machining flaws that may have a pronounced effect on flexural strength. Machining damage imposed during specimen preparation can be either a random interfering factor or an inherent part of the strength characteristic to be measured. Surface preparation can also lead to residual stresses. Universal or standardized test methods of surface preparation do not exist. It should be understood that final machining steps may or may not negate machining damage introduced during the early coarse or intermediate machining.

5.3 Slow crack growth can lead to a rate dependency of flexural strength. The testing rate specified in this standard may or may not produce the inert flexural strength whereby negligible slow crack growth occurs. See Method C 1368.

6. Apparatus

6.1 *Loading*—Specimens may be force in any suitable testing machine provided that uniform rates of direct loading can be maintained. The force measuring system shall be free of initial lag at the loading rates used and shall be equipped with a means for retaining readout of the maximum force as well as a force-time or force-deflection record. The accuracy of the testing machine shall be in accordance with Practices E 4.⁸

6.2 *Four-Point Flexure Four-Point- 1/4 Point Fixtures* (Fig. 1), having support spans as given in Table 1.

6.3 *Three-Point Flexure Three-Point Fixtures* (Fig. 1), having a support span as given in Table 1.

6.4 *Bearings*, three- and four-point flexure.

6.4.1 Cylindrical bearings shall be used for support of the test specimen and for load application. The cylinders may be made of a ceramic with an elastic modulus between 200 and 400 GPa (30 to 60 × 10⁶ psi) and a flexural strength no less than 275 MPa (≈40 ksi). The loading cylinders must remain elastic (and have no plastic deformation) over the load and temperature ranges used, and they must not react chemically with or contaminate the test specimen. The test fixture shall also be made of a ceramic that is resistant to permanent deformation.

6.4.2 The bearing cylinder diameter shall be approximately 1.5 times the beam depth of the test specimen size used (see Table 2).

⁸ The accuracy requirement is different from that specified in Test Method C 1161 and is a concession to difficulties incurred in conducting elevated temperature testing. The accuracy required by Practices E 4 is 1 %; Test Method C 1161 calls for 0.5 %.

⁷ The boldface numbers in parentheses refer to the list of references at the end of the text.

TABLE 2 Nominal Bearing Diameters

Configuration	Diameter, mm
A	2.0 to 2.5
B	4.5
C	9.0

TABLE 3 Specimen Sizes

Configuration	Width (b), mm	Depth (d), mm	Length (L_T), mm, min
A	2.0	1.5	25
B	4.0	3.0	45
C	8.0	6.0	90

6.4.3 The bearing cylinders shall be positioned carefully such that the spans are accurate to within ± 0.10 mm. The load application bearing for the three-point configurations shall be positioned midway between the support bearings within ± 0.10 mm. The load application (inner) bearings for the four-point configurations shall be centered with respect to the support (outer) bearings within ± 0.10 mm.

6.4.4 The bearing cylinders shall be free to rotate in order to relieve frictional constraints (with the exception of the middle-load bearing in three-point flexure, which need not rotate). This can be accomplished as shown in Fig. 2 and Fig. 3. Annex A2 illustrates the action required of the bearing cylinders. Note

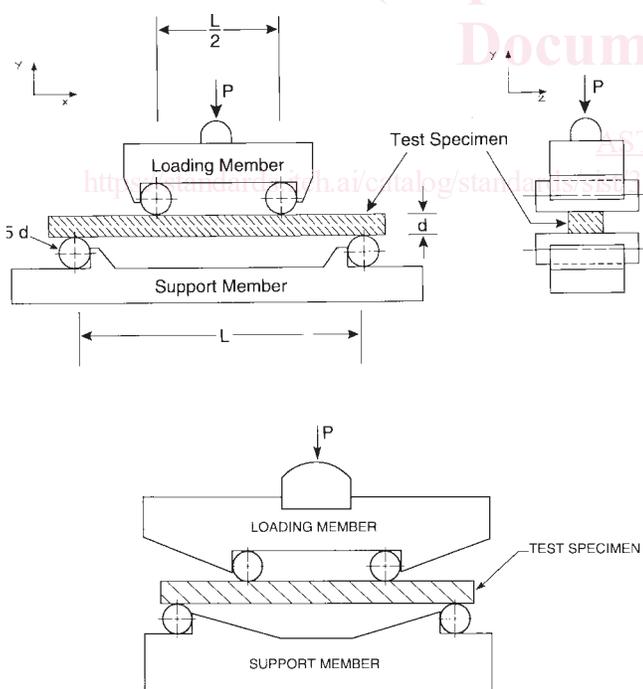
that the outer-support bearings roll outward and the inner-loading bearings roll inward.⁹

6.5 *Semiarticulating Four-Point Fixture*—Specimens prepared in accordance with the parallelism requirements of 7.1 may be tested in a semiarticulating fixture as illustrated in Fig. 2 and in Fig. A2.1(a). All four bearings shall be free to roll. The two inner bearings shall be parallel to each other to within 0.015 mm over their length. The two outer bearings shall be parallel to each other to within 0.015 mm over their length. The inner bearings shall be supported independently of the outer bearings. All four bearings shall rest uniformly and evenly across the specimen surfaces. The fixture shall be designed to apply equal load to all four bearings.

6.6 *Fully Articulating Four-Point Fixture*—Specimens that are as-fired, heat treated, or oxidized often have slight twists or unevenness. Specimens that do not meet the parallelism requirements of 7.1 shall be tested in a fully articulating fixture as illustrated in Fig. 3 and in Fig. A2.1(b). Well-machined specimens may also be tested in fully-articulating fixtures. All four bearings shall be free to roll. One bearing need not articulate. The other three bearings shall articulate to match the specimen's surface. All four bearings shall rest uniformly and evenly across the specimen surfaces. The fixture shall apply equal load to all four bearings.

6.7 *Semiarticulated Three-Point Fixture*—Specimens prepared in accordance with the parallelism requirements of 7.1 may be tested in a semiarticulating fixture as illustrated in Fig. A2.2(a). The middle bearing shall be fixed and not free to roll. The two outer bearings shall be parallel to each other to within 0.015 mm over their length. The two outer bearings shall articulate together to match the specimen surface, or the middle bearing shall articulate to match the specimen surface. All three bearings shall rest uniformly and evenly across the specimen surface. The fixture shall be designed to apply equal load to the two outer bearings.

6.8 *Fully Articulated Three-Point Flexure*—Specimens that do not meet the parallelism requirements of 7.1 shall be tested in a fully-articulating fixture as illustrated in Figs. A2.2(b) or A2.2(c). Well-machined specimens may also be tested in fully-articulating fixtures. The two support (outer) bearings shall be free to roll outward. The middle bearing shall not roll. Any two of the bearings shall be capable of articulating to match the specimen surface. All three bearings shall rest



NOTE 1—Configuration:

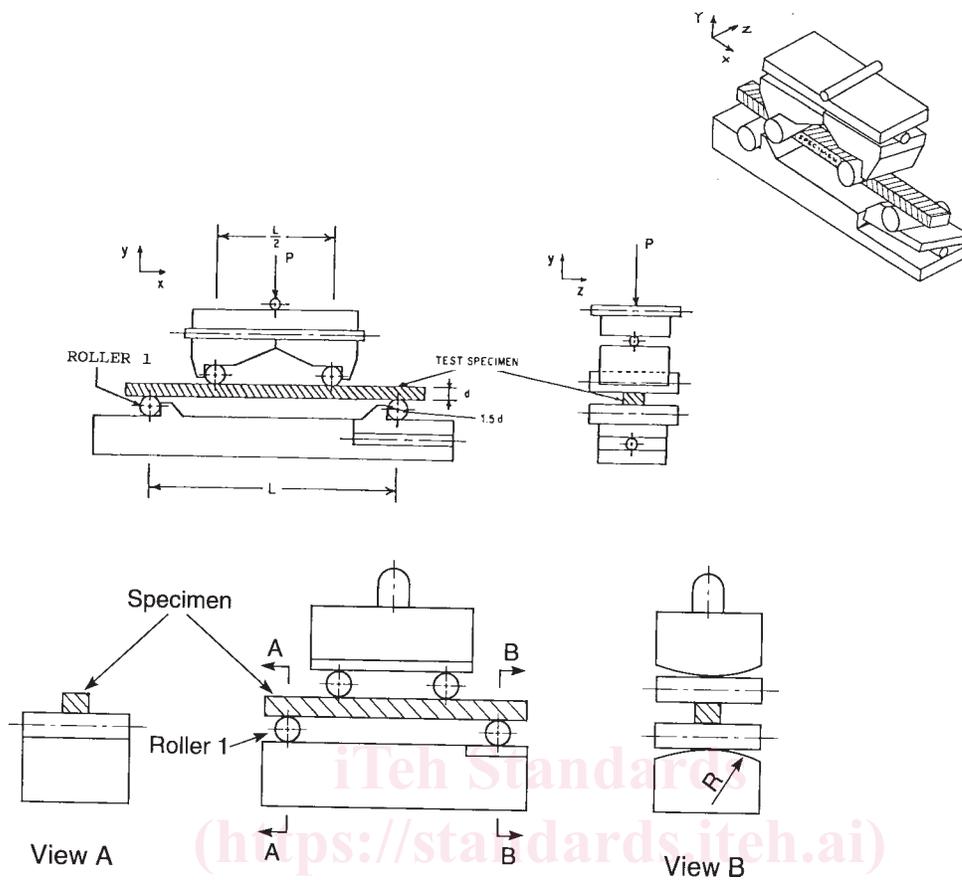
- A: L = 20 mm
- B: L = 40 mm
- C: L = 80 mm

FIG. 2 Schematics of Semiarticulated Four-Point Fixtures Suitable for Flat and Parallel Specimens; Load is Applied Through a Rounded and Well-Centered Tip that Permits the Loading Member to Tilt as Necessary to Ensure Uniform Loading

⁹ In general, fixed-pin fixtures have frictional constraints that can cause a systematic error on the order of 5 to 15 % in flexure strength (see Refs (1, 2, 4 to 7)). Since this error is systematic (constant for all specimens in a sample), it will lead to a bias in estimates of the mean strength and will shift a Weibull curve a fixed amount of stress. The scatter, however, will remain constant.

Rolling-pin fixtures are required by this test method. It is recognized that they may not be feasible in some instances, in which case fixed-pin fixtures may be used, but this must be stated explicitly in the report, and justification must be given as noted in 10.1.16.

Some fixtures have loading cylinders that fit into square slots with a slight clearance. Of course, the clearance must be such that the possible spans are within the prescribed limits of this test method. Unfortunately, for any given test, it is usually not possible to ascertain whether a roller rests against an inner or outer shoulder, and thus it is possible that some rollers may be free to roll and others not. This can lead to the superimposition of a random error on the results. Such fixtures should therefore be used with caution.



NOTE 1—Configuration:

- A: L = 20 mm
- B: L = 40 mm
- C: L = 80 mm

FIG. 3 Schematics of Fully Articulating Four-Point Fixtures Suitable for Twisted or Uneven Specimens; One of the Four Load Bearings (for Example, Roller No. 1) Should Not Articulate About the x Axis. The Other Three Will Provide the Necessary Degrees of Freedom. The Radius R in the Bottom Fixture Should Be Sufficiently Large Such that Contact Stresses on the Roller are Minimized.

uniformly and evenly across the specimen surface. The fixture shall be designed to apply equal load to the two outer bearings.

6.9 *System Compliance*—The compliance of the load train shall be characterized for the loading range used and the testing temperature.¹⁰ The load train and fixtures shall be sufficiently rigid so that at least 80 % of the crosshead motion is transmitted to the actual test specimens. The load train and fixtures shall not permanently deform during testing. It is not necessary to check the system compliance for every test sequence, provided that it has been characterized previously for the identical setup.

¹⁰ Compliance can be measured by inserting an oversized block onto the flexure fixture, loading it to the maximum expected break force at the test temperature, and recording a load-deflection graph. The block must be a ceramic material that will remain elastic under these conditions. The compliance check shall be made with the entire force train in place, especially the load bearing rollers. It is recommended that the block be at least five times thicker than the normal test specimen and one to two times thicker than the normal specimen width.

6.10 *Fixture Material*, essentially inert for the testing conditions used. The fixture shall be oxidation resistant if the testing is performed in air.¹¹

6.11 *Heating Apparatus*—A furnace capable of meeting the following requirements:

6.11.1 The furnace shall be capable of establishing and maintaining a constant temperature during each testing period.

¹¹ Various grades of silicon carbide are available that will be suitable for fixtures and load trains. Hot-pressed or sintered silicon carbides with low additive content are elastic to temperatures in excess of 1500°C. Siliconized silicon carbides and high-purity aluminas are less expensive and are available in a variety of shapes, but they exhibit creep deformations at temperatures above 1200°C. Recrystallized silicon carbides are elastic to temperatures up to 2000°C but are relatively weak due to their porosity. Graphites are extremely refractory but are restricted to usage in inert atmospheres. They may suffice for load rams or portions of fixtures, but they should be avoided for use where there are concentrated loads, such as loading bearings, since graphite is too soft. Avoid materials that will oxidize significantly at test temperatures (if testing in air) or that will react chemically with or contaminate test specimens.

is used, for example, as-cast, sintered, or injection-molded parts. No additional machining specifications are relevant. An edge chamfer is not necessary in this instance. As-fired specimens are especially prone to twist or warpage and may not meet the parallelism requirements. A fully articulating fixture (see 6.6 and Fig. 3) shall be used in this instance.

7.2.2 Application-Matched Machining—The specimen shall be given the same surface preparation as that given to a component. Unless the process is proprietary, the report shall be specific concerning the stages of material removal, wheel grits, wheel bonding, and the amount of material removed per pass.

7.2.3 Customary Procedure—This procedure shall be used in instances in which a customary machining procedure has been developed that is completely satisfactory for a class of materials (that is, it induces no unwanted surface damage or residual stresses). It shall be fully specified in the report.

7.2.4 Standard Procedure—In the instances in which 7.2.1 through 7.2.3 are not appropriate, 7.2.4.1 through 7.2.4.4 shall apply. This procedure shall serve as minimum requirements, and a more stringent procedure may be necessary.

7.2.4.1 All grinding shall be performed with an ample supply of appropriate filtered coolant to keep the workpiece and wheel constantly flooded and the particles flushed. Grinding shall be performed in at least two stages, ranging from coarse to fine rates of material removal. All machining shall be performed in the surface grinding mode and shall be parallel to the specimen long axis, as shown in Fig. 5. No Blanchard or rotary grinding shall be used.

7.2.4.2 The stock removal rate shall not exceed 0.03 mm (0.001 in.) per pass to the last 0.06 mm (0.002 in.) per face. Final (and intermediate) finishing shall be performed with a diamond wheel that is between 320 and 500 grit. No less than 0.06 mm per face shall be removed during the final finishing phase, and at a rate of not more than 0.002 mm (0.0001 in.) per pass. Remove approximately equal stock from opposite faces. Machine all four long faces this way. The two end faces need not be machined as carefully as the long faces.

7.2.4.3 Materials with low fracture toughness and a greater susceptibility to grinding damage may require finer grinding wheels at very low removal rates.

7.2.4.4 The four long edges of each specimen shall be chamfered uniformly at 45°, a distance of 0.12 ± 0.03 mm, as shown in Fig. 4. Alternatively, they can be rounded with a radius of 0.15 ± 0.05 mm. Edge finishing shall be comparable

to that applied to the specimen surfaces. In particular, the direction of machining shall be parallel to the specimen long axis. If chamfers are larger than the tolerance allows, corrections shall be made to the stress calculation according to Refs (1, 2). Alternatively, a chamfer is not required if a specimen can be prepared with an edge free of machining damage.

7.2.5 Handling Precautions—Exercise care in the storing and handling of specimens to avoid the introduction of random and severe flaws, such as might occur if the specimens were allowed to impact or scratch each other.

7.3 Number of Specimens—A minimum of 10 specimens shall be required for the purpose of estimating the mean. A minimum of 30 shall be necessary if estimates regarding the form of the strength distribution are to be reported (for example, a Weibull modulus). The number of specimens required by this test method has been established with the intent of determining not only reasonable confidence limits on strength distribution parameters, but also to help discern multiple-flaw population distributions. More than 30 specimens are recommended if multiple-flaw populations are present.

8. Procedure

8.1 Test the specimens on their appropriate fixture in specific testing configurations. Test the Size A specimens on either the four-point A fixture or the three-point A fixture. Similarly, test B specimens on B fixtures and C specimens on C fixtures. A fully articulated fixture is required if the specimen parallelism requirements cannot be met.

8.2 Specimens may be loaded into either a cold furnace, with the whole system then heated to operating temperature, as specified in 8.3 or directly into a hot furnace as specified in 8.4. Do not mark load points since the marker material could react chemically with the specimen. (Ordinary pencil lead is graphite bonded by a clay. The clay can melt or react with a ceramic.)

8.3 Cold-Furnace Loading—Specimens may be loaded onto the test fixtures in a cooled furnace. Some means of ensuring alignment of the system during subsequent heating to test temperature shall be provided. The furnace shall then be raised to the test temperature at a constant heating rate that shall be stated in the report. Temperature overshoot (over the test temperature) shall be strictly controlled and shall be no more than 5°C. The temperature shall be held constant (soak time) for the necessary time for the specimen and furnace to come to equilibrium. The soak time shall be stated in the report.

8.4 Hot-Furnace Loading—Alternatively, specimens may be loaded directly into a hot furnace. This shall be conducted in a fashion so as to minimize or eliminate thermal shock damage to the specimen. Temperature overshoot (over the test temperature) shall be strictly controlled and shall be no more than 5°C. The temperature shall be held constant (soak time) for the necessary time for the specimen and furnace to come to equilibrium. The soak time shall be stated in the report.^{18,19,20,21}

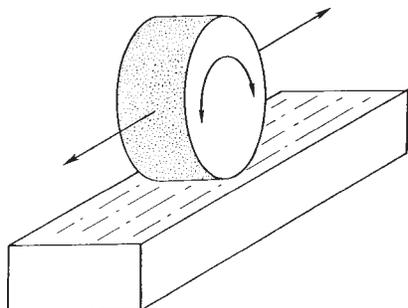


FIG. 5 Surface Grinding Parallel to the Specimen Longitudinal Axis

¹⁸ The fixtures may be either left in the furnace the entire time or removed partially or completely, depending on the details of the system.