Designation: F 15 – 98

Standard Specification for Iron-Nickel-Cobalt Sealing Alloy [1]

This standard is issued under the fixed designation F 15; 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 specification covers an iron-nickel-cobalt alloy, UNS K94610 containing nominally 29 % nickel, 17 % cobalt, and 53 % iron, in the forms of wire, rod, bar, strip, sheet, and tubing, intended primarily for sealing to glass in electronic applications.

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

1.3 The following hazard caveat pertains only to the test method portion, Sections 13 and 14 of this specification. *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:*

E 3 Methods of Preparation of Metallographic Specimens[2]

E 8 Test Methods of Tension Testing of Metallic Materials[2]

E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials[2]

E 112 Test Methods for Determining Average Grain Size[2]

E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer[3]

F 14 Practice for Making and Testing Reference Glass-Metal Bead-Seal[4]

F 140 Practice for Making Reference Glass-Metal Butt Seals and Testing for Expansion Characteristics by Polarimetric Methods[4]

F 144 Practice for Making Reference Glass-Metal Sandwich Seal and Testing for Expansion Characteristics by Polarimetric Methods[4]

3. Ordering Information

3.1 Orders for material under this specification shall include the following information:

3.1.1 Size,

3.1.2 Temper (Section 6),

3.1.3 Surface finish (Section 10),

3.1.4 Marking and packaging (Section 17), and

3.1.5 Certification if required.

4. Chemical Requirements

4.1 The material shall conform to the requirements as to chemical composition prescribed in Table 1.

5. Surface Lubricants

5.1 All lubricants used during cold-working operations, such as drawing, rolling, or spinning, shall be capable of being removed readily by any of the common organic degreasing solvents.

6. Temper

6.1 The desired temper of the material shall be specified in the purchase order.

6.2 *Tube*—Unless otherwise agreed upon by the supplier or manufacturer and the purchaser, these forms shall be given a final bright anneal by the manufacturer and supplied in the annealed temper.

6.3 *Strip and Sheet*— These forms shall be supplied in one of the tempers given in Table 2 or in deep-drawing temper, as specified.

6.4 *Wire and Rod*— These forms shall be supplied in one of the tempers given in Table 3 as specified. Unless otherwise specified, the material shall be bright annealed and supplied in temper A (annealed).

7. Grain Size

7.1 Strip and sheet for deep drawing shall have an average grain size not larger than ASTM No. 5 (Note 1), and no more than 10 % of the grains shall be larger than No. 5 when measured in accordance with Test Methods E 112.

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[1] This specification is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.03 on Metallic Materials.

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[2] *Annual Book of ASTM Standards*, Vol 03.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 15.02.

TABLE 1 Chemical Requirements

Element	Composition, %
Iron, nominal	53 [A]
Nickel, nominal	29 [A]
Cobalt, nominal	17 [A]
Manganese, max	0.50
Silicon, max	0.20
Carbon, max	0.04
Aluminum, max	0.10 [B]
Magnesium, max	0.10 [B]
Zirconium, max	0.10 [B]
Titanium, max	0.10 [B]
Copper, max	0.20
Chromium, max	0.20
Molybdenum, max	0.20

[A] The iron, nickel, and cobalt requirements listed are nominal. They shall be adjusted by the manufacturer so that the alloy meets the requirements for coefficient of thermal expansion given in Table 4.

[B] The total of aluminum, magnesium, zirconium, and titanium shall not exceed 0.20 %.

TABLE 2 Tensile Strength Requirements for Sheet and Strip

Temper Designation	Temper Name	Tensile Strength, ksi(MPa)
A	annealed	82 max (570 max)
B	¼ hard	75 to 90 (520 to 630)
C	half hard	85 to 100 (590 to 700)
D	¾ hard	95 to 110 (660 to 770)
E	hard	100 min (700 min)

TABLE 3 Tensile Strength Requirements for Wire and Rod

Temper Designation	Tensile Strength, ksi (MPa)
A	85 (585) max
B	85 to 105 (585 to 725)
C	95 to 115 (655 to 795)
D	105 to 125 (725 to 860)
E	125 (860) min

NOTE 1—This corresponds to a grain size of 0.065 mm, or 16 grains/in.^{2} of image at 100 × .

8. Hardness

8.1 *Deep-Drawing Temper*—For deep drawing, the hardness shall not exceed 82 HRB for material 0.100 in. (2.54 mm) and less in thickness and 85 HRB for material over 0.100 in. in thickness when determined in accordance with Test Methods E 18.

8.2 *Rolled and Annealed Tempers*—Hardness tests when properly applied can be indicative of tensile strength. Hardness scales and ranges for these tempers, if desirable, shall be negotiated between supplier and purchaser.

9. Tensile Strength

9.1 *Sheet and Strip*:

9.1.1 Tensile strength shall be the basis for acceptance or rejection for the tempers given in Table 2 and shall conform with the requirements prescribed.

9.1.2 Tension test specimens shall be taken so the longitudinal axis is parallel to the direction of rolling and the test shall be performed in accordance with Test Methods E 8.

9.2 *Wire and Rod*:

9.2.1 Tensile strength shall be the basis for acceptance or rejection for the tempers given in Table 3 and shall conform to the requirements prescribed.

9.2.2 The test shall be performed in accordance with Test Method E 8.

10. Surface Finish

10.1 The standard surface finishes available shall be those resulting from the following operations:

10.1.1 Hot rolling,

10.1.2 Forging,

10.1.3 Centerless grinding (rod),

10.1.4 Belt polishing,

10.1.5 Cold rolling, and

10.1.6 Wire drawing.

11. Thermal Expansion Characteristics

11.1 The average linear coefficients of thermal expansion shall be within the limits specified in Table 4.

12. Test for Thermal Expansion

12.1 Heat the specimen in a hydrogen atmosphere for 1 h at 900°C, followed by 15 min at 1100°C. Between the 900 and 1100°C heat-treatment periods, the specimen may be cooled to room temperature if desired. Cool the specimen from 1100 to 200°C in the hydrogen atmosphere at a rate not to exceed 5°C/min.

12.2 Determine the thermal expansion characteristics in accordance with Test Method E 228.

NOTE 2—For critical glass sealing applications, it is recommended that the user conduct functional testing in accordance with Practices F 14, F 140 or F 144. Such tests circumvent possible problems with thermal expansion measurements and glass setting point estimates.

13. Transformation

13.1 The temperature of the gamma-to-alpha transformation shall be below −78.5°C when the material is tested in accordance with Section 14. However, for material whose smallest dimension is over ⅞ in. (22.2 mm), some localized transformation, acceptable to the purchaser, may be tolerated.

14. Test for Transformation

14.1 Cut the specimen from any part of the material, but preferably including the entire cross section, degrease it, then heat treat it as described in 12.1. When cool, polish the cross section of the specimen and etch (Note 3) it in accordance with Method E 3. Then subject the specimen to the temperature produced by an excess of dry ice in acetone (−78.5°C) for at least 4 h. After the low-temperature treatment, examine the specimen at a mangification of 150× for the presence of the acicular crystals characteristic of the alpha phase. Because

TABLE 4 Coefficients of Thermal Expansion

Temperature Range, °C	Average Linear Coefficient of Thermal Expansion, [A] µm/m·°C
30 to 400	4.60 to 5.20
30 to 450	5.10 to 5.50

[A] Typical thermal expansion data for the alloy covered by these specifications are provided in Appendix X1.

these crystals may occur only in small localized areas, examine carefully the entire polished cross section.

14.2 Specimens that show no transformation and that show partial transformation are illustrated in Fig. 1 and Fig. 2, respectively.

NOTE 3—A suggested etchant is a solution of three parts by volume of concentrated hydrochloric acid and one part of concentrated nitric acid saturated with cupric chloride ($CuCl_2 \cdot 2H_2O$). This etchant is more effective when allowed to stand for 20 min after mixing. After several hours it loses its strength and should be discarded at the end of the day. Etching is best accomplished by swabbing the specimen with cotton soaked with the etchant. Etching is usually complete when the surface of the metal appears to have turned dull.

15. Dimensions and Permissible Variations

15.1 *Cold-Rolled Strip*—Cold-rolled strip shall conform to the permissible variations in dimensions prescribed in Table 5, Table 6, and Table 7.

15.2 *Round Wire and Rod*—Wire and rod shall conform to the permissible variations in dimensions prescribed in Table 8.

15.3 *Cold-Drawn Tubing*—Cold-drawn tubing, available either as seamless or welded, shall conform to the permissible variations prescribed in Table 9.

16. General Requirements

16.1 The material shall be commercially smooth, uniform in cross section, in composition, and in temper; it shall be free of scale, corrosion, cracks, seams, scratches, slivers, and other defects as best commercial practice will permit.

17. Packaging and Marking

17.1 Packaging shall be subject to agreement between the purchaser and the seller.

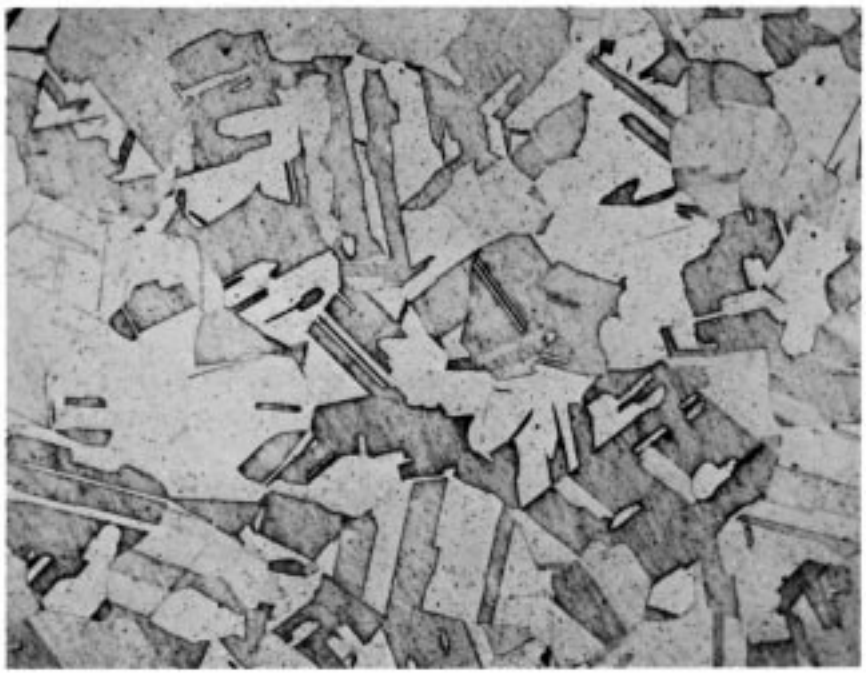
17.2 The material as furnished under this specification shall be identified by the name or symbol of the manufacturer and by melt number. The lot size for determining compliance with the requirements of this specification shall be one heat.

18. Investigation of Claims

18.1 Where any material fails to meet the requirements of this specification, the material so designated shall be handled in accordance with a mutual agreement between the purchaser and the seller.

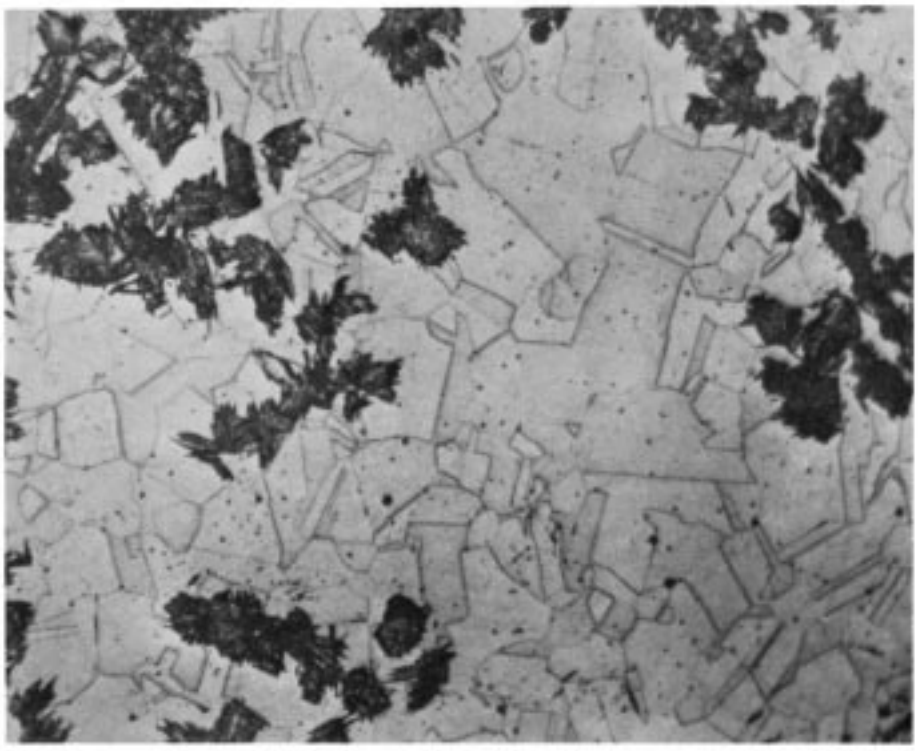
19. Keywords

19.1 controlled expansion alloy; glass to metal sealing; iron-nickel-cobalt alloy; UNS #K94610; vacuum electronic applications



150×

FIG. 1 Normal Annealed Specimen Showing No Transformation



150×

FIG. 2 Partially Transformed Specimen

TABLE 5 Permissible Variations in Thickness of Cold-Rolled Strip

NOTE 1— Measurement shall be made at least ⅜ in. (9.5 mm) from the edge of strip over 1 in. (25.4 mm) wide.

Specified Thickness, in. (mm)	Permissible Variations in Thickness for Width Given, ± in. (mm)			
	Under 3 (76)	Over 3 to 6 (76 to 152)	Over 6 to 12 (152 to 305)	Over 12 to 16 (305 to 406)
0.160 to 0.100 (4.06 to 2.54), incl	0.002 (0.051)	0.003 (0.076)	0.004 (0.102)	0.004 (0.102)
0.099 to 0.069 (2.51 to 1.75), incl	0.002 (0.051)	0.003 (0.076)	0.003 (0.076)	0.004 (0.102)
0.068 to 0.050 (1.73 to 1.27), incl	0.002 (0.051)	0.003 (0.076)	0.003 (0.076)	0.003 (0.076)
0.049 to 0.035 (1.24 to 0.89), incl	0.002 (0.051)	0.0025 (0.064)	0.003 (0.076)	0.003 (0.076)
0.034 to 0.029 (0.86 to 0.74), incl	0.0015 (0.038)	0.002 (0.051)	0.0025 (0.064)	0.0025 (0.064)
0.028 to 0.026 (0.71 to 0.66), incl	0.0015 (0.038)	0.0015 (0.038)	0.002 (0.051)	0.002 (0.051)
0.025 to 0.020 (0.64 to 0.51), incl	0.001 (0.025)	0.0015 (0.038)	0.002 (0.051)	0.002 (0.051)
0.019 to 0.017 (0.48 to 0.43), incl	0.001 (0.025)	0.001 (0.025)	0.0015 (0.038)	0.002 (0.051)
0.016 to 0.012 (0.41 to 0.31), incl	0.001 (0.025)	0.001 (0.025)	0.0015 (0.038)	0.0015 (0.038)
0.011 to 0.0101 (0.28 to 0.26), incl	0.001 (0.025)	0.001 (0.025)	0.001 (0.025)	0.0015 (0.038)
0.010 to 0.0091 (0.25 to 0.23), incl	0.001 (0.025)	0.001 (0.025)	0.001 (0.025)	0.001 (0.025)
0.009 to 0.006 (0.23 to 0.15), incl	0.00075 (0.019)	0.00075 (0.019)	...	...
Under 0.006 (0.15)	0.0005 (0.013)	0.0005 (0.013)	...	...

TABLE 6 Permissible Variations in Thickness Across Width of Strip

Specified Thickness		Maximum Variation in Thickness Across Width of Strip, Within Those Provided for in Table 4 for Edge Measurements for Widths and Thicknesses Given, in. (mm)					
in.	mm	5 (127) and Under		Over 5 to 12 (127 to 300)		Over 12 to 24 (300 to 600), incl	
		in.	mm	in.	mm	in.	mm
0.005 to 0.010, incl	0.17 to 0.03, incl	0.00075	0.0191	0.001	0.025	0.0015	0.038
Over 0.010 to 0.025, incl	0.03 to 0.06, incl	0.001	0.025	0.0015	0.038	0.002	0.051
Over 0.025 to 0.065, incl	0.06 to 0.16, incl	0.0015	0.038	0.002	0.051	0.0025	0.064
Over 0.065 to 3/16, excl	0.16 to 0.48, excl	0.002	0.051	0.0025	0.064	0.003	0.076

TABLE 7 Permissible Variations in Width of Cold-Rolled Strip Supplied in Coils

Specified Thickness, in. (mm)	Permissible Variations in Width for Widths Given, ± in. (mm)					
	Under ½ to 3/16 (12.7 to 4.8)	½ to 6 (12.7 to 152)	Over 6 to 9 (152 to 229)	Over 9 to 12 (229 to 305)	Over 12 to 20 (305 to 508)	Over 20 to 23 15/16 (508 to 608)
0.187 to 0.161 (4.75 to 4.09)	...	0.016 (0.41)	0.020 (0.51)	0.020 (0.51)	0.031 (0.79)	0.031 (0.79)
0.160 to 0.100 (4.06 to 2.54)	0.010 (0.25)	0.010 (0.25)	0.016 (0.41)	0.016 (0.41)	0.020 (0.51)	0.020 (0.51)
0.099 to 0.069 (2.51 to 1.75)	0.008 (0.20)	0.008 (0.20)	0.010 (0.25)	0.010 (0.25)	0.016 (0.41)	0.020 (0.51)
0.068 (1.73) and under	0.005 (0.13)	0.005 (0.13)	0.005 (0.13)	0.010 (0.25)	0.016 (0.41)	0.020 (0.51)

TABLE 8 Permissible Variations in Diameter of Wire and Rod

Specified Diameter, in. (mm)		Permissible Variations in Diameter, ± in. (mm)	
Wire (Coiled, Spooled or Straight Lengths)			
0.002 to 0.0043	(0.05 to 0.110)	0.0002	(0.005)
0.0044 to 0.0079	(0.111 to 0.202)	0.00025	(0.006)
0.008 to 0.0149	(0.20 to 0.379)	0.0003	(0.008)
0.015 to 0.0199	(0.38 to 0.507)	0.0004	(0.010)
0.020 to 0.0309	(0.51 to 0.786)	0.0005	(0.013)
0.031 to 0.0409	(0.79 to 1.04)	0.0006	(0.015)
0.041 to 0.0609	(1.04 to 1.548)	0.0007	(0.018)
0.061 to 0.0809	(1.55 to 2.056)	0.0008	(0.020)
0.081 to 0.1259	(2.06 to 3.199)	0.001	(0.025)
0.126 to 0.1569	(3.20 to 3.99)	0.0015	(0.038)
0.157 to 0.250	(4.00 to 6.35)	0.002	(0.051)
Rod, Centerless Ground Finish (Straight Lengths)			
0.030 to 0.0549	(0.76 to 1.396)	0.0005	(0.013)
0.055 to 0.1249	(1.40 to 3.174)	0.001	(0.035)
0.125 to 0.499	(3.18 to 12.70)	0.0015	(0.038)
0.500 to 0.999	(12.7 to 25.37)	0.002	(0.051)
1.000 to 1.625	(25.4 to 41.28)	0.0025	(0.064)
1.626 to 1.749	(41.30 to 44.40)	0.003	(0.08)
1.750 to 1.999	(44.45 to 50.77)	0.004	(0.10)
2.000 to 4.000	(50.80 to 101.60)	0.005	(0.13)

TABLE 9 Permissible Variations in Dimensions of Standard Tubing

Specified Outside Diameter, in. (mm)	Permissible Variations [A]		
	Outside Diameter, in. (mm)	Inside Diameter, in. (mm)	Wall Thickness, ± %
Under 0.093 (2.36)	+ 0.002 (0.05) − 0.000	+ 0.000 −0.002 (0.05)	10
0.093 to 0.187 (2.36 to 4.76), excl	+ 0.003 (0.08) − 0.000	+ 0.000 −0.003 (0.08)	10
0.187 to 0.500 (4.76 to 12.70), excl	+ 0.004 (0.10) − 0.000	+ 0.000 − 0.004 (0.10)	10
0.500 to 1.500 (12.70 to 38.10), excl	+ 0.005 (0.13) − 0.000	+ 0.000 −0.005 (0.13)	10

[A] Any two of the three dimensional tolerances listed may be specified.

APPENDIX

(Nonmandatory Information)

X1.

X1.1 *Coefficient of Thermal Expansion (CTE) at Elevated Temperatures*— For various applications, the high-temperature CTE is required for the alloy defined by this specification. The data provided in Table X1.1 are for material produced in the early 1970s.

X1.2 *On-Cooling Data from 1000°C to –268°C, Using 30°C as Reference Temperature* —The CTE data in Table X1.2 is provided by a producer of the F-15 alloy.

X1.3 *Statistical Information on CTE Requirements as Supplied by Materials Producers*—Two producers of the alloy defined by this specification have provided statistical information regarding the CTE requirements defined in Table X1.3. Producer A provided both average CTE and associated standard deviation for an unspecified number of heats, which it had produced during the past several years. All of this information has been generated in the on-heating mode. That information is shown in Table X1.4. Producer B provided histogram information showing the distribution of CTE values, obtained in the on-cooling mode, for both of the temperature ranges (30–400°C and 30–450°C) required in Table X1.3. This information covers heats that has beenproduced and determined to conform to this specification in the past several years. That information is shown in Table X1.3 and Table X1.5.

TABLE X1.1 Average CTE to Elevated Temperatures (On-Heating Data[A])

Temperature Range, °C	Average Linear Coefficient of Thermal Expansion µm/m–°C
30 to 100	5.8
30 to 150	5.6
30 to 200	5.4
30 to 250	5.3
30 to 300	5.1
30 to 350	4.9
30 to 400	4.8
30 to 450	5.2
30 to 500	6.1
30 to 550	6.8
30 to 600	7.5
30 to 650	8.2
30 to 700	8.7
30 to 750	9.3
30 to 800	9.8
30 to 850	10.3
30 to 900	10.8
30 to 950	11.2
30 to 1000	11.7

[A] This data was obtained from Bertolotti R. L., "Thermal Expansions of Kovar and Ceramvar and Seals of These Materials to Alumina," SAND 74-8003, Sandia National Laboratories, September 1974. Data presented by Bertolotti have been obtained on heating using a special dilatometer, which could operate from –180°C up to 1000°C.

TABLE X1.2 Coefficient of Thermal Expansion to Both Elevated and Cryogenic Temperatures (On-Cooling Data)

Temperature Range, °C	Average Linear Coefficient of Thermal Expansion µm/m –°C
30 to–268	4.9
30 to–196	6.1
30 to–163	6.4
30 to–100	6.5
30 to–78	6.5
100 to 30	6.3
200 to 30	5.7
300 to 30	5.2
400 to 30	5.0
450 to 30	5.3
500 to 30	6.1
600 to 30	7.8
700 to 30	8.9
800 to 30	10.1
900 to 30	11.3
1000 to 30	12.2

TABLE X1.3 Producer B Information on 30–400°C CTE Data (On-Cooling Data[A])

Range of CTE (µm/m –°C)	Frequency of Occurrence
4.60–4.70	0.045
4.70–4.80	0.100
4.80–4.90	0.175
4.90–5.00	0.230
5.00–5.10	0.180
5.10–5.20	0.270

[A] The average of this data is 4.97 (µm/m –°C).

TABLE X1.4 Statistical Information Provided by Producer A (On-Heating Data)

Temperature Range, °C	Average CTE (µm/m –°C)	Standard Deviation
30 to 400	4.92	0.13
30 to 450	5.27	0.12

TABLE X1.5 Producer B Information on 30–450°C CTE Data (On-Cooling Data[A])

Range of CTE (µm/m –°C)	Frequency of Occurrence
5.10–5.20	0.225
5.20–5.30	0.190
5.30–5.40	0.330
5.40–5.50	0.255

[A] The average of this data is 5.31 (µm/m –°C).