

### Standard Specification for Iron-Nickel-Cobalt Sealing Alloy<sup>1</sup>

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 ( $\epsilon$ ) 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: <sup>2</sup>
- E 3 Methods of Preparation of Metallographic Specimens
- E 8 Test Methods of Tension Testing of Metallic Materials
- E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials
- E 92 Test Method for Vickers Hardness of Metallic Materials
- E 112 Test Methods for Determining Average Grain Size
- E 140 Standard Hardness Conversion Tables for Metals
- E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer
- F 14 Practice for Making and Testing Reference Glass-Metal Bead-Seal

- F 140 Practice for Making Reference Glass-Metal Butt Seals and Testing for Expansion Characteristics by Polarimetric Methods
- F 144 Practice for Making Reference Glass-Metal Sandwich Seal and Testing for Expansion Characteristics by Polarimetric Methods

#### 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).

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<sup>&</sup>lt;sup>1</sup> This specification is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.03 on Metallic Materials.

Current edition approved May 1, 2004. Published June 2004. Originally approved in 1961 as F15-61T. Last previous edition approved in 1998 as F15-98.

<sup>&</sup>lt;sup>2</sup> 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.

**TABLE 1** Chemical Requirements

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

<sup>A</sup> 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.

<sup>*B*</sup> 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)
А	annealed	82 max (570 max)
В	1/4 hard	75 to 90 (520 to 630)
С	half hard	85 to 100 (590 to 700)
D	3/4 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
В	85 to 105 (585 to 725)
С	95 to 115 (655 to 795)
D	105 to 125 (725 to 860)
E	125 (860) min

#### 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.

Note 1—This corresponds to a grain size of 0.065 mm, or 16 grains/in.² of image at 100  $\times$  .

#### 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. See also Test Method E 92 for Vickers Hardness and Table 3, E 140 for the appropriate conversion between various hardness scales.

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.

NOTE 3—The thermal treatment described in this section is for purposes of the thermal expansion test only. Consult the non-mandatory appendix of this document for guidance on annealing conditions for various product forms.

#### 13. Transformation

13.1 The temperature of the gamma-to-alpha transformation shall be below  $-78.5^{\circ}$ C when the material is tested in accordance with Section 14. However, for material whose smallest dimension is over  $7/_{8}$  in. (22.2 mm), some localized transformation, acceptable to the purchaser, may be tolerated.

TABLE 4 Coefficients of Thermal Expansion

Temperature Range, °C	Average Linear Coefficient of Thermal Expansion, <sup>A</sup> μm/m.°C
30 to 400	4.60 to 5.20
30 to 450	5.10 to 5.50

<sup>A</sup> Typical thermal expansion data for the alloy covered by these specifications are provided in Appendix X1.

NOTE 4—Lower transformation temperatures, ranging to as low as  $-196^{\circ}$ C, may be negotiated between supplier and purchaser. The  $-196^{\circ}$ C transformation temperature corresponds to immersing a sample (prepared according to 14.1) in liquid nitrogen for a minimum of 1 h.

#### 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 5) it in accordance with Method E 3. Then subject the specimen to the temperature produced by an excess of dry ice in acetone ( $-78.5^{\circ}$ C) for at least 4 h. After the low-temperature treatment, examine the specimen at a mangification of  $150 \times$  for the presence of the acicular crystals characteristic of the alpha phase. Because 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 5—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.

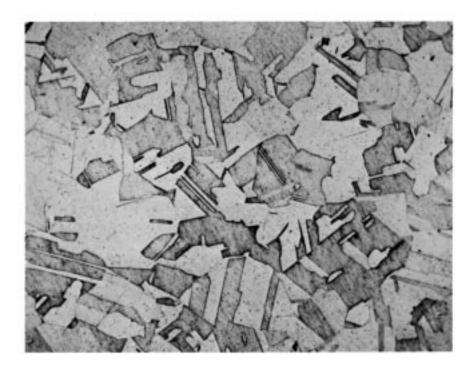
17.2 The material as furnished under this specification shall be identified by the name or symbol of the manufacturer and by melt number. The lot size for determining compliance with the requirements of this specification shall be one heat.

#### 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

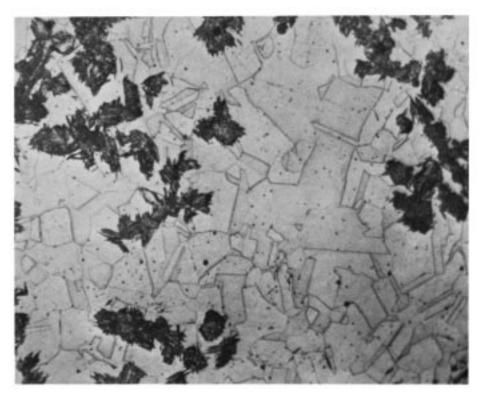
19.1 controlled expansion alloy; glass to metal sealing; iron-nickel-cobalt alloy; UNS #K94610; vacuum electronic applications



**150**×

FIG. 1 Normal Annealed Specimen Showing No Transformation

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**150**×

FIG. 2 Partially Transformed Specimen

NOTE 1- Measurement shall be made at least 3/8 in. (9.5 mm) from the edge of strip over 1 in. (25.4 mm) wide.

Creatived Thickness in (mm)	Permissible Variations in Thickness for Width Given, $\pm$ in. (mm)				
Specified Thickness, 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)			

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#### TABLE 6 Permissible Variations in Thickness Across Width of Strip

S	pecified Thickness				Vidth of Strip, Witl /idths and Thickne		
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

		Permis	sible Variations in Widt	h for Widths Given, $\pm$ in	n. (mm)	
Specified Thickness, in. (mm)	Under ½ to ¾16 (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 <sup>15</sup> ⁄ <sub>16</sub> (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) 0.068 (1.73) and under	0.008 (0.20) 0.005 (0.13)	0.008 (0.20) 0.005 (0.13)	0.010 (0.25) 0.005 (0.13)	0.010 (0.25) 0.010 (0.25)	0.016 (0.41) 0.016 (0.41)	0.020 (0.51) 0.020 (0.51)

#### TABLE 8 Permissible Variations in Diameter of Wire and Rod

Specified Diameter, in. (mm)			ons in Diameter, ± in. nm)
	Wire (Coiled, Spooled or Str	aight 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 <sup>A</sup>		
Specified Outside Diameter, in. (mm)	Outside Diameter, in. (mm)	Inside Diameter, in. (mm)	Wall Thickness, $\pm$ %
Under 0.093 (2.36)	+ 0.002 (0.05)	+ 0.000	10
	- 0.000	-0.002 (0.05)	
0.093 to 0.187 (2.36 to 4.76), excl	+ 0.003 (0.08)	+ 0.000	10
	- 0.000	-0.003 (0.08)	
0.187 to 0.500 (4.76 to 12.70), excl	+ 0.004 (0.10)	+ 0.000	10
	- 0.000	- 0.004 (0.10)	
0.500 to 1.500 (12.70 to 38.10), excl	+ 0.005 (0.13)	+ 0.000	10
	- 0.000	-0.005 (0.13)	

 $^{\scriptscriptstyle A} \, {\rm Any}$  two of the three dimensional tolerances listed may be specified.

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#### APPENDIX

#### (Nonmandatory Information)

#### X1. Detailed Thermal Expansion Data; Annealing Conditions and Grain Growth in Piece Parts and Components

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. It is important to note that the CTE values cited are for annealed temper material.

X1.2 On-Cooling Data from  $1000^{\circ}$ C to  $-268^{\circ}$ C, Using  $30^{\circ}$ 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 have been produced and determined to conform to this specification in the past several years. That information is shown in Table X1.3 and Table X1.5.

X1.4 Annealing Temperatures Recommended for Various Product Forms of the F15 Alloy—The following section presents typical annealing temperatures for specific product

 TABLE X1.1 Average CTE to Elevated Temperatures (On-Heating Data<sup>A</sup>)

	,
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

<sup>A</sup>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.

and oryogenic reinperatures (on-oboling 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 X1.2 Coefficient of Thermal Expansion to Both Elevated and Crvogenic Temperatures (On-Cooling Data)

TABLE X1.3	Producer	<b>B</b> Information	on 30-400°C	CTE Data (On-
		Cooling Dat	a <sup>A</sup> )	

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

<sup>A</sup> The average of this data is 4.97 ( $\mu$ m/m –°C).

TABLE X1.4 S	tatistical 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>B</b> Information	on 30-450°C	CTE Data (On-
		Cooling Dat	ta <sup>A</sup> )	

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

<sup>A</sup> The average of this data is 5.31 ( $\mu$ m/m –°C).

forms, at the piece part or component level, made from the F15 alloy. The intent is to help the user avoid conditions where excessive grain growth could render material unfit for specific applications.

X1.4.1 Annealing Temperatures for F15 Alloy Lead Wire— Table X1.6 shows the results of of study of grain growth in lead wire material. Two types of wire were examined: a 0.018 in. diameter size wire, procured in the cold worked condition, and a 0.020 in. diameter size wire, procured in the mill annealed condition. All samples were annealed in a wet hydrogen

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Material Condition	ASTM Grain Size Number for Mill Annealed Material (Range of ASTM Grain Size Numbers Based on Log- Normal Analysis)	Mill Annealed Material: Knoop Mlcrohardness (50 or 100 g load, as indicated)	Grain Size Number for Cold Worked Material (Range of ASTM Grain Size Numbers Based on Log-Normal Analysis)	Cold Worked Material: Knoop Microhardness (50 or 100 g load, as indicated)
Starting Condition	> 9.0	161 (±9.9) 5 g	(N/A-cold worked condition)	254 (±6.5) 50 g
900°C, 1 h, Wet Hydrogen Atmosphere	7.7 (11.1-5.9)	162 (±1.2) 50 g 160. (±2.1) 100 g	7.9 (10.0-6.4	153 (±2.5) to g 156. (±4.9) 100 g
1000°C, 1 h, Wet Hydrogen Atmosphere	6.3 (9.0-4.3)	152 (±2.2) 50 g 153 (±1.5) 100 g	6.1 (9.8-4.2)	152 (±1.3) 50 g 151. (±1.5) 100 g
1100°Ċ, 1 h, Wet Hydrogen Atmosphere	5.0 (7.5-3.4)	150. (±0.5) 50 g 151 (±1.2) 100 g	4.3 (7.3-2.5)	149 (±1.0) 50 g 152 (±1.8) 100 g

<sup>A</sup> Hardness data represent the average of 10 indentations. The "range of grain size numbers" represents the intercept lengths in the range between the 10 and 90 percentiles, respectively, based on the log-normal distribution.

TABLE X1.7 Effect of Isothermal Annealing Cycle (Argon Atmosphere) on Grain Growth and Microhardness of 0.018 in. diameter F15
Alloy Lead Wire

Material Condition	Range of ASTM Grain Size Numbers for Mill Annealed Material	Mill Annealed Material: Knoop Mlcrohardness (100 g load)	Range of ASTM Grain Size Numbers for Cold Worked Material	Cold Worked Material: Knoop Microhardness ( 100 g load)
Starting Condition	8-9	177 ± 2.8	(N/A-cold worked condition)	282 (±5.4)
900°C, 1 h, Argon Atmosphere	4-8	162 ± 9.2	6-8	163.9 ± 8.1
1000°C, 1 h, Argon Atmosphere	4-6	159 ± 9.2	5-7	159.9 ± 7.6
1100°C, 1 h, Argon Atmosphere	1-5	$155~\pm~5.0$	0-4	154.8 ± 8.2

atmosphere. Knoop hardness values (50 g or 100 g loads) for both types of material are shown in Table  $X1.7.^3$ 

X1.4.2 Additional data, supplied by a materials producer of the F15 alloy, are shown in Table X1.7. Their study examined the effect of the same thermal processes, using an Argon atmosphere, on the grain size and Knoop microhardness. Thus, a direct comparison could be made with the data in Table X1.6. Both cold worked and mill annealed material were examined. The 0.018 in. diameter wire was processed using typical fabrication processing to obtain wire.

X1.4.3 The data shown in both Table X1.6 and Table X1.7 indicate that annealing process cycles in excess of 1000°C, 1 h, will lead to significant grain growth in lead wire. The 1100°C, 1 h, anneal produces coarser grain sizes that should be avoided, if possible. It should be noted that there are some applications (for example, when brazing with OFHC Copper) that necessitate 1100°C process cycles. In these cases, it is important to minimize the total time spent in excess of 1050°C in order to avoid excessive grain coarsening.

<sup>&</sup>lt;sup>3</sup> Further details on this study can be found in the proceedings paper: Stephens, J. J., Greulich, F. A., and Beavis, L. C., , "High Temperature Grain Growth and Oxidation of Fe-29Ni-17Co (Kovar<sup>®</sup>) Alloy Leads," published as pages 79–112 in the book *Low Thermal Expansion Alloys and Composites*, Stephens, J. J., and Frear, D. R., eds., TMS, Warrendale, PA, 1994.

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