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Standard Specification for Iron-Nickel-Cobalt Alloys for Metal-to-Ceramic Sealing Applications ¹

This standard is issued under the fixed designation F 1466; 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 two iron-nickel-cobalt alloys, the former, (UNS No. K94630), containing nominally 29 % nickel, 17 % cobalt, and 53 % iron, the latter, (UNS No. K94620), nominally 27 % nickel, 25 % cobalt and 48 % iron, in the forms of wire, rod, bar, strip, sheet, and tubing, intended primarily for brazed metal-to-ceramic seals with alumina ceramics, for vacuum electronic applications. Unless otherwise indicated, all articles apply to both alloys.
- 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 14 and 16 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:
- D 1971 Practices for Digestion of Samples for Determination of Metals by Flame Atomic Absorption or Plasma Emission Spectroscopy²
- E 3 Methods of Preparation of Metallographic Specimens³
- E 8 Test Methods for Tension Testing of Metallic Materials³
- E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials³
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁴
- E 45 Practice for Determining the Inclusion Content of Steel³
- E 92 Test Method for Vickers Hardness of Metallic Materials³
- E 112 Test Methods for Determining Average Grain Size³

- E 140 Standard Hardness Conversion Tables for Metals³ E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer⁴
- E 354 Test Methods for Chemical Analysis of High-Temperature, Electrical, Magnetic and Other Similar Iron, Nickel and Cobalt Alloys⁵
- E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, Oxygen and Hydrogen in Steel and in Iron, Nickel and Cobalt Alloys⁶
- E 1060 Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis⁶
- F 15 Specification for Iron-Nickel-Colbalt Sealing Alloy⁷

3. Ordering Information

- 3.1 Orders for material under this specification shall include the following information:
 - 3.1.1 Alloy, as indicated with UNS number,
 - 3.1.2 Size,
 - 3.1.3 Temper designation (Section 6),
 - 3.1.4 Surface finish (Section 10),
 - 3.1.5 Marking and packaging (Section 19), and
- 3.1.6 Certification, if required. Please note that certification should include traceability of the heat to the original manufacturer.

4. Chemical Requirements

4.1 Each alloy 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 between the supplier or the manufacturer and the purchaser, these forms shall

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² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 03.01.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Annual Book of ASTM Standards, Vol 03.05.

⁶ Annual Book of ASTM Standards, Vol 03.06.

⁷ Annual Book of ASTM Standards, Vol 10.04.

TABLE 1 Chemical Requirements

Note 1—Round observed or calculated values to the nearest unit in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of Practice E 29.

| Element | UNS No. K94630 | UNS No. K94620 |
|-----------------|------------------------|------------------------|
| Iron, nominal | remainder ^A | remainder ^A |
| Nickel, nominal | 29 ^A | 27 ^A |
| Cobalt, nominal | 17 ^A | 25 ^A |
| Manganese, max | 0.35 | 0.35 |
| Silicon, max | 0.15 | 0.15 |
| Carbon, max | 0.02 | 0.02 |
| Aluminum, max | 0.01 ^B | 0.01 ^B |
| Magnesium, max | 0.01 ^B | 0.01 ^B |
| Zirconium, max | 0.01 ^B | 0.01 ^B |
| Titanium, max | 0.01 ^B | 0.01 ^B |
| Copper, max | 0.20 | 0.20 |
| Chromium, max | 0.03 | 0.03 |
| Molybdenum, max | 0.06 | 0.06 |
| Phosphorus, max | 0.006 ^C | 0.006 ^C |
| Sulfur, max | 0.006 ^C | 0.006 ^C |

^A The iron, nickel, and cobalt requirements are nominal and may be adjusted by the manufacturer to meet the requirements for the coefficient of thermal expansion as specified in 12.1.

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).

Note 1—For rod forms, air anneal, followed by centerless grinding to remove scale, is an acceptable alternate.

7. Grain Size

7.1 Strip and sheet for deep drawing shall have an average grain size not larger than ASTM No. 5 (Note 2), and no more than 10 % of the grains shall be larger than No. 5 when measured in accordance with Test Methods E 112.

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

7.2 Finer grain sizes for deep drawing quality shall be negotiated between user and supplier.

8. Hardness

8.1 Deep-Drawing Temper—For deep drawing, the hard-

TABLE 2 Tensile Strength Requirements for Strip and Sheet

| Temper | Temper | Tensile Strength, ksi (MPa) | | |
|-------------|-----------|-----------------------------|------------------------------------|--|
| Designation | · | UNS No. K94630 | UNS No. K94620 (Nominal Values) | |
| A | annealed | 82 max (565 max) | 85 max (586 max) | |
| В | 1/4 hard | 75 to 90 (517 to 621) | 85 to 100 (586 to 689) | |
| С | half hard | 85 to 100 (586 to 689) | 95 to 110 (655 to 758) | |
| D | 3/4 hard | 95 to 110 (655 to 758) | 105 to 120 (724 to 827) | |
| E | hard | 100 min (689 min) | 120 min (827 min) | |

TABLE 3 Tensile Strength Requirements for Wire and Rod

| Tompor | Tensile Strength, ksi (MPa) | | |
|-----------------------|-----------------------------|------------------------------------|--|
| Temper Designation | UNS No. K94630 | UNS No. K94620 (Nominal Values) | |
| Α | 85 (586) max | 85 (586) max | |
| В | 85 to 105 (586 to 724) | 85 to 100 (586 to 689) | |
| С | 95 to 115 (655 to 793) | 95 to 110 (655 to 758) | |
| D | 105 to 125 (724 to 862) | 105 to 120 (724 to 827) | |
| E | 125 (862) min | 120 (827) min | |

ness 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 Testing and tables in E 140.

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 Strip and Sheet:
- 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 Methods 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 and rod drawing.

11. Inclusion Content

11.1 Wire, Rod, Bar, Strip and Sheet—These product forms shall be free of inclusions, cracks, blow holes and other defects that are detrimental to the quality of subsequent product. The maximum inclusion rating number shall be 2 for Inclusion Types, A, B, C and D in both the thin and heavy series shown in Plate I using Practice E 45, Method A, Worst-Field Technique.

Note 3—The test for inclusions may be performed on billet sections. In such cases, the sample section must include regions that correspond to the top of the ingot.

Note 4—Product section size information at which the inclusion ratings were taken should be included.

12. Thermal Expansion Characteristics

12.1 The average linear coefficients of thermal expansion

 $^{^{\}it B}$ The total of aluminum, magnesium, titanium, and zirconium shall not exceed 0.04 %.

^C The total of phosphorus and sulfur shall not exceed 0.010. %.

shall be within the limits specified in Table 4.

13. Test for Thermal Expansion

- 13.1 Heat the specimen in a non-oxidizing atmosphere for 1 h at 1000°C. Cool at a rate not to exceed 300°C per hour.
- 13.2 Determine the thermal expansion characteristics in accordance with Test Method E 228.

14. Transformation

- 14.1 (UNS No. K94630 only) The temperature of the gamma-to-alpha transformation shall be below -78.5° C when the material is tested in accordance with Section 16. However, for material whose smallest dimension is over $\frac{7}{8}$ in. (22.2 mm), some localized transformation, acceptable to the purchaser, may be tolerated.
- 14.2 The temperature of the gamma-alpha transformation for UNS No. K94620 is well below 78.5°C. Tests for transformation in this alloy, if necessary, shall be negotiated between supplier and purchaser.
- 14.3 For alloy UNS No. K94630, consult the nonmandatory appendix of Specification F 15 for applicable thermal expansion information based on producer heat data and recommended thermal expansion values over a wide range of temperatures.

15. Test for Transformation

15.1 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 13.1. When cool, polish the cross section of the specimen and etch (Note 5) it in accordance with Methods E 3. Then subject the specimen to the temperature produced by an excess of dry ice in methanol (–78.5°C) for at least 4 h. After the low-temperature treatment, examine the specimen at a magnification of 150× 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.

Note 5—A suggested etchant is a solution of three parts by volume of

TABLE 4 Coefficients of Thermal Expansion

| Temperature Range,° C | Average Linear Coefficient of Thermal Expansion, μm/m.°C | | |
|-----------------------|--|------------------------------------|--|
| | UNS No. K94630 | UNS No. K94620 (Nominal Values) | |
| 30–400 | 4.60-5.20 | | |
| 30-450 | 5.10-5.50 | | |
| 30-500 | | 7.30-7.90 | |
| 30–600 | | 8.00-8.60 | |

concentrated hydrochloric acid and one part of concentrated nitric acid saturated with cupric chloride (CuCl $_2$ ·2H $_2$ O). 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.

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

16. Chemical Analysis

- 16.1 This describes the chemical analysis techniques to be used in case of dispute. Wherever applicable, the analysis procedures described in Practices D 1971, Test Methods E 354, E 1019 and Practice E 1060 should be utilized.
 - 16.2 Do chemical analysis as follows:
 - 16.2.1 Carbon, Sulfur, Combustion method.
- 16.2.2 Aluminum, Chromium, Magnesium—Atomic absorption method.
- 16.2.3 All Other Elements Shown in Table 1 (excluding Iron, Nickel and Cobalt)—Atomic absorption, optical emission or inductively coupled plasma (ICP or ICAP) methods.

Note 6—The iron, nickel and cobalt requirements are nominal (see Table 1).

17. Dimensions, Mass, and Permissible Variations

- 17.1 *Cold-Rolled Strip*—Cold-rolled strip shall conform to the permissible variations in dimensions prescribed in Table 5, Table 6, and Table 7.
- 17.2 Round Wire and Rod—Wire and rod shall conform to the permissible variations in dimensions prescribed in Table 8.
- 17.3 *Cold-Drawing Tubing*—Cold-drawn tubing, available either as seamless or welded, shall conform to the permissible variations prescribed in Table 9.

18. General Requirements

18.1 The material supplied under this specification 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.

19. Packaging and Package Marking

- 19.1 Packaging shall be subject to agreement between the purchaser and the seller.
- 19.2 The material as furnished under this specification shall be identified by the name or symbol of the manufacturer and by heat number. The lot size for determining compliance with the requirements of this specification shall be one heat.

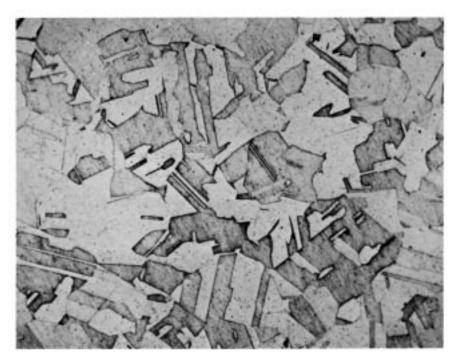


FIG. 1 Normal Annealed Specimen Showing No Transformation

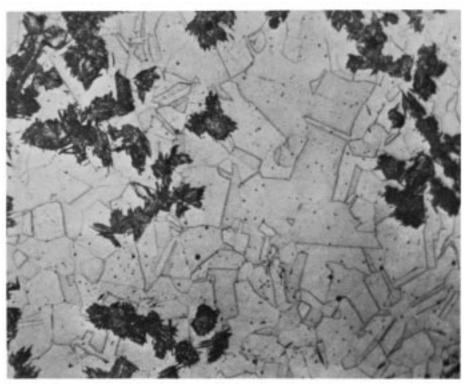


FIG. 2 Partially Transformed Specimen

20. Investigation of Claims

20.1 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.

21. Keywords

21.1 control of thermal expansion; iron-nickel-cobalt alloys; metal/ceramic brazing; oxide-forming impurities and inclusions; UNS No. K94620; UNS No. K94630; vacuum electronic applications

TABLE 5 Permissible Variations in Thickness of Cold-Rolled Strip

| Considered This leaves in (common | 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- 0.100 (4.06- 2.54), incl | 0.002 (0.051) | 0.003 (0.076) | 0.004 (0.102) | 0.004 (0.102) | |
| 0.099- 0.069 (2.51- 1.75), incl | 0.002 (0.051) | 0.003 (0.076) | 0.003 (0.076) | 0.004 (0.102) | |
| 0.068- 0.050 (1.73- 1.27), incl | 0.002 (0.051) | 0.003 (0.076) | 0.003 (0.076) | 0.003 (0.076) | |
| 0.049- 0.035 (1.24- 0.89), incl | 0.002 (0.051) | 0.0025 (0.064) | 0.003 (0.076) | 0.003 (0.076) | |
| 0.034- 0.029 (0.86- 0.74), incl | 0.0015 (0.038) | 0.002 (0.051) | 0.0025 (0.064) | 0.0025 (0.064) | |
| 0.028- 0.026 (0.71- 0.66), incl | 0.0015 (0.038) | 0.0015 (0.038) | 0.002 (0.051) | 0.002 (0.051) | |
| 0.025- 0.020 (0.64- 0.51), incl | 0.001 (0.025) | 0.0015 (0.038) | 0.002 (0.051) | 0.002 (0.051) | |
| 0.019- 0.017 (0.48- 0.43), incl | 0.001 (0.025) | 0.001 (0.025) | 0.0015 (0.038) | 0.002 (0.051) | |
| 0.016- 0.012 (0.41- 0.31), incl | 0.001 (0.025) | 0.001 (0.025) | 0.0015 (0.038) | 0.0015 (0.038) | |
| 0.011- 0.0101 (0.28- 0.26), incl | 0.001 (0.025) | 0.001 (0.025) | 0.001 (0.025) | 0.0015 (0.038) | |
| 0.010- 0.0091 (0.25- 0.23), incl | 0.001 (0.025) | 0.001 (0.025) | 0.001 (0.025) | 0.001 (0.025) | |
| 0.009- 0.006 (0.23- 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, in. (mm) | | 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) | | | |
|--|-------------------|---|----------------------------------|--|--|
| | 5 (127) and Under | Over 5 to 12 (127 to 300) | Over 12 to 24 (300 to 600), incl | | |
| 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

| | | Permi | ssible Variations in V | Vidth for Widths Give | n, ± in. (mm) | |
|-------------------------------|--|-------------------------|-----------------------------|---------------------------|-------------------------------|--|
| Specified Thickness, in. (mm) | Under ½ to ¾ 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) |
| 0.187-0.161 (4.75-4.09) | | 0.016 (0.42) | 0.020 (0.51) | 0.020 (0.51) | 0.031 (0.79) | 0.031 (0.79) |
| 0.160-0.100 (4.06-2.54) | 0.010 (0.25) | 0.010 (0.25) | 0.016 (0.41) | 0.016 (0.41) | 0.020 (0.51) | 0.020 (0.51) |
| 0.099-0.069 (2.51-1.75) | 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 Variation | ons in Diameter, \pm in. (mm) |
|--|---------------------------|-------------------------|---------------------------------|
| Wire (Coiled, Spooled or Straight Lengths) | | | |
| 0.002-0.0043 | (0.05–0.110) | 0.0002 | (0.005) |
| 0.0044-0.0079 | (0.111-0.202) | 0.00025 | (0.006) |
| 0.008-0.0149 | (0.20-0.379) | 0.0003 | (0.008) |
| 0.015-0.0199 | (0.38–0.507) | 0.0004 | (0.010) |
| 0.020-0.0309 | (0.51–0.786) | 0.0005 | (0.013) |
| 0.031-0.0409 | (0.79–1.04) | 0.0006 | (0.015) |
| 0.041-0.0609 | (1.04–1.548) | 0.0007 | (0.018) |
| 0.061-0.0809 | (1.55-2.056) | 0.0008 | (0.020) |
| 0.081-0.1259 | (2.06-3.199) | 0.001 | (0.025) |
| 0.126-0.1569 | (3.20-3.99) | 0.0015 | (0.038) |
| 0.157-0.250 | (4.00–6.35) | 0.002 | (0.051) |
| | Rod, Centerless Ground Fi | nish (Straight Lengths) | |
| 0.030-0.0549 | (0.76–1.396) | 0.0005 | (0.013) |
| 0.055-0.1249 | (1.40–3.174) | 0.001 | (0.035) |
| 0.125-0.499 | (3.18–12.70) | 0.0015 | (0.038) |
| 0.500-0.999 | (12.7–25.37) | 0.002 | (0.051) |
| 1.000-1.625 | (25.4–41.28) | 0.0025 | (0.064) |
| 1.626-1.749 | (41.30–44.40) | 0.003 | (0.08) |
| 1.750-1.999 | (44.45–50.77) | 0.004 | (0.10) |
| 2.000-4.000 | (50.80-101.60) | 0.005 | (0.13) |



TABLE 9 Permissible Variations in Dimensions of Standard Tubing

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

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