

Designation: F1466 - 99 (Reapproved 2005)

Standard Specification for Iron-Nickel-Cobalt Alloys for Metal-to-Ceramic Sealing Applications¹

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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:²
- D1971 Practices for Digestion of Water Samples for Determination of Metals by Flame Atomic Absorption, Graphite Furnace Atomic Absorption, Plasma Emission Spectroscopy, or Plasma Mass Spectrometry
 - E3 Guide for Preparation of Metallographic Specimens
 - E8 Test Methods for Tension Testing of Metallic Materials
 - E18 Test Methods for Rockwell Hardness of Metallic Materials
 - E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- 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 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.

- E45 Test Methods for Determining the Inclusion Content of Steel
- E92 Test Method for Vickers Hardness of Metallic Materials
- E112 Test Methods for Determining Average Grain Size
- E140 Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, and Scleroscope Hardness
- E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer
- E354 Test Methods for Chemical Analysis of High-Temperature, Electrical, Magnetic, and Other Similar Iron, Nickel, and Cobalt Alloys
- E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques
- E1060 Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis

3. Ordering Information

- 3.1 Orders for material under this specification shall include the following information: 9698/astm-11466-992005
 - 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.

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 E29.

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.

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 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 E112.

TABLE 2 Tensile Strength Requirements for Strip and Sheet

Temper Designation	Temper Name	Tensile Strength, ksi (MPa)	
		UNS No. K94630	UNS No. K94620 (Nominal Values)
Α	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)	
A	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	

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 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 E18. See also Test Method E92 for Vickers Hardness Testing and tables in E140.
- 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 E8.
 - 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 E8.

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

 $^{^{\}it B}$ 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. %

Types, A, B, C and D in both the thin and heavy series shown in Plate I using Practice E45, 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 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 E228.

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 E92 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 E3. 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.

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	

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.

15.2 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 D1971, Test Methods E3, E1019 and Practice E1060 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.

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.