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NICKEL-BERYLLIUM ALLOY AND METHOD OF HEAT TREATING SAME

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This invention relates to nickel-based beryllium-containing alloys capable of being readily machined by standard techniques.

More particularly, the invention relates to such alloys and to a method of heat treating the same to provide high tensile strength and hardness.

Commercial nickel-based beryllium-containing alloys have many applications where a material having high strength and hardness, oxidation and corrosion resistance, good thermal conductivity, good wear resistance at both room and elevated temperatures is required. Articles from these alloys are generally formed by casting techniques. Cast bodies are usually annealed, machined to desired shapes, and then heat treated to develop a high degree of strength and hardness.

In such applications, various machining operations are generally required. However, because articles produced from such commercial alloys are difficult to machine, the applications are much more limited than would otherwise be the case in the absence of this problem.

It is, therefore, an object of the invention to provide nickel-based beryllium-containing alloys which are capable of being readily machined by standard production techniques.

A further object is to provide such a readily machinable alloy which has tensile strength, hardness, and other properties comparable to those of commercially available alloys of approximately the same nickel and beryllium content.

A still further object is to provide a method of heat treating such alloys to induce the desired tensile strength and hardness properties.

In accordance with these and other objects and advantages hereinafter set forth, the invention comprises uniformly dispersing, in a nickel-beryllium alloy, small graphite nodules by incorporating, in a melt of the alloy, a sufficient amount of both carbon and an agent selected from the group consisting of magnesium, calcium and cerium to effect nodularization of the carbon. Alloy bodies formed from the melt are heat treated by heating at 1950° F. until the beryllium is in solid solution with the nickel matrix, by quenching the alloy, by reheating the quenched alloy to 900 to 1000° F. until a predetermined tensile strength is developed and cooling to room temperature.

The following compositions are illustrative of the alloys of the invention. Elements such as molybdenum, titanium, tungsten, niobium, tantalum, zirconium, vanadium, cobalt and iron may be added to improve elevated temperature strength, grain refinement and other properties.

ALLOY NO. 1

	Broad Range, Percent	Preferred Range, Percent
Beryllium	1.0–3.0	2.00–2.25
Carbon	0.35–1.30	0.50–0.75
Magnesium	0.10–1.35	0.15–0.50
Nickel	Balance	

ALLOY NO. 2

	Broad Range, Percent	Preferred Range, Percent
Beryllium	1.0–3.0	2.00–2.25
Carbon	0.35–1.30	0.50–0.75
Calcium	0.05–0.50	0.20–0.30
Nickel	Balance	

ALLOY NO. 3

	Broad Range, Percent	Preferred Range, Percent
Beryllium	1.0–3.0	2.00–2.25
Carbon	0.35–1.30	0.50–0.75
Cerium	0.10–0.20	0.10–0.15
Nickel	Balance	

As a substitute for cerium, misch metal in double the amount and, alternatively, lanthanum or neodymium in equal quantities, can be used.

Functioning of the alloying constituents

Generally, beryllium is employed in a range from about 1 to about 3%, since less than 1% beryllium content does not provide for sufficient tensile strength and hardness for commercial applications. More than 3% beryllium causes objectionable brittleness and increases the cost of the alloy.

Carbon content of 0.35% and higher (in combination with the nodularizing agent) results in the formation of graphite nodules which, if uniformly dispersed and of small size, improves the ease of machining these alloys while not significantly affecting the strength and hardness. The preferred carbon content of 0.5 to 0.75% (in combination with the nodularizing agent) results in small uniformly dispersed nodules and an alloy of improved machinability and one, which when properly heat treated, has high strength and hardness. A carbon content of less than 0.35% (in combination with the nodularizing agent) fails to produce the desired nodularization and accompanying improved machinability. With a carbon content of more than 1.5% (in combination with the nodularizing agent), large graphite nodules are formed and the hardness and strength of the alloys, when heat treated, are much lower than when the carbon is maintained in the preferred range. Magnesium, calcium and cerium are added, in combination with carbon in the ranges specified, to produce the formation of nodular or spherical particles of graphite instead of the flake form thereof. The flake form of graphite, which exists when a sufficient quantity of nodularizing agents are not present, although enhancing machinability of the commercial alloys, lowers their tensile strength and hardness; whereas, the presence of graphite in nodular form improves machinability of the commercial alloys with little sacrifice of their tensile strength and hardness properties. Obviously, higher amounts of the nodularizing agent metals are required when the carbon content is increased.

Examples illustrative of the invention are listed below in three sections. First, the compositions, preparation, strength and hardness microstructure and estimated machinability of several Ni-Be-C-Mg alloys are given in Table I. For comparative purposes, the strength and hardness, microstructure and machinability of both a commercial alloy, as well as a graphitized alloy without the nodularizing agent of the invention, are also included in Table I. Second, the compositions, preparation, strength

and hardness, micro-hardness and estimated machinability of several Ni-Be-C-Ca and Ni-Be-C-Ce alloys are presented in Table II. Finally, actual machinability data collected during the evaluation of representative alloys of the invention is presented.

Criteria established herein for commercial usefulness in nickel-base beryllium-containing alloys are preferably a minimum tensile strength of 150,000 p.s.i. and, specifically, a minimum hardness of Rc45 when properly heat treated. For rating as a "readily machinable" nickel-beryllium alloy, a demonstration in the annealed condition of at least twice the machinability of commercial alloys in the annealed condition, as measured by a standard tool life test, was required in the estimated machinability evaluations.

To avoid extensive, tedious and expensive machinability evaluation, the microstructures developed in the alloys of this invention were used to estimate relative machinability properties. A trained metallurgist can readily and effectively interpret the relative machinability of these alloys by the size, shape and distribution of graphite inclusions. Such inclusions serve both as a chip breaker and solid lubricant in machining operations and thus reduce power requirements and lengthen tool life. The microstructure observations listed in Tables I and II were made by a trained metallurgist and are considered valid, as such appraisal is generally accepted in the field of metallurgical science.

Preparation, processing and metallurgical evaluation of Ni-Be-C-Mg alloys

Heats of Ni-Be-C alloys were made by melting in air using a 3,000-cycle induction furnace of 35-lb. capacity and lined with magnesia. Charges consisted of sheared, electrolytic nickel squares, nickel-beryllium master alloy of 50/50 composition, and solid, dry graphite. The nickel was melted and heated to 250° F. and the melt "killed" with a small addition of Ni-Be master. The required amount of graphite was added by stirring until it dissolved into the melt. The remainder of the Ni-Be master was added and the melt temperature lowered to 2525° F. The Mg was added as Ni–13 to 16% Mg master alloy and stirred in just before pouring. The melts were poured into standard 0.505" reduced diameter tensile bar cavities in phosphate bonded sand molds. The tensile bar castings and metallographic specimens were trimmed down from the gates and sprues. All specimens were heat treated as required and machined and prepared for evaluation. Furnace annealed specimens were heated to 1950° F. for three hours in a large muffle furnace and then slowly cooled to room temperature with the furnace turned off. The solution annealed specimens were similarly heated to 1950° F. for three hours, but then quenched into cold water. Machining operations to prepare tensile, hardness and metallographic samples were all of a conventional nature and consisted in the main of sawing and turning. Aged specimens were initially solution annealed and then heated to 950° F. for three hours and air cooled. A minimum two-fold improvement in machinability over commercial alloys was observed while preparing metallurgical evaluation specimens from annealed alloys of the invention.

TABLE I. MECHANICAL PROPERTIES AND SUMMARY OF MICROSTRUCTURE FOR VARIOUS Ni-Be-C HEATS
[All material in solution annealed and aged condition (AT) unless otherwise specified]

Example	Chemical Composition in Percent				Tensile Properties UTS in K s.i.	Percent El.	Hardness Rc	Microstructure at 100X, Relative Estimated Machinability and Relative Strength and Hardness
	Be	Mg	C	Ni				
	Commercial Alloy							
1	2.19		0.09	Bal.	223.0	4.0	52	Carbon all in solid solution; no visible graphite. Poor machinability but maximum strength and hardness.
	Commercial Alloy with Carbon added but no Nodularizing Agent							
2	2.56	0	0.81	Bal.	110.5	1.5	40	Large graphite flakes. Machinability excellent but strength and hardness unsatisfactorily low.
	Alloys with 0.6% C—Variable Mg Content							
3 (a)	2.17	0.44	0.63	Bal.			25	As cast—Uniformly distributed graphite nodules of 0.010 to 0.030 mm. dia.
(b)	2.17	0.44	0.63	Bal.			22	Solution Annealed—Microstructure similar to Example 3 (a) above.
(c)	2.17	0.44	0.63	Bal.	195.6	0.6	51	Solution Annealed and Aged—Microstructure similar to Examples 3 (a) and 3 (b) above but with graphite nodules of 0.005 to 0.30 mm. in dia. Machinability improved more than twice that of Example #1. Strength and hardness satisfactorily high.
4	2.34	0.85	0.58	Bal.	157.6	0.9	51	Graphite nodules not as well distributed and somewhat larger than those in Example 3 (c). Machinability somewhat better than Example 3 (c). Strength marginal but hardness satisfactory.
5	2.41	1.30	0.61	Bal.	133.2	1.0	52.5	Fewer but larger graphite nodules than in Example #4, approximately 0.060 mm. in dia. Machinability somewhat better than Example #4. Strength poor but hardness satisfactory.
	Alloys with 1.0% C—Variable Mg Content							
6	2.15	0.41	0.98	Bal.	188.2	0.8	50.5	Well dispersed graphite nodules about 0.010 mm. in dia. Machinability greater than two times that of commercial alloy Example #1. Strength and hardness are high.
7	2.32	0.84	0.98	Bal.	207.1	1.0	51	Graphite nodules well dispersed and about 0.030 mm. in dia. Machinability somewhat greater than that of Example #6. Strength and hardness are high.
8	2.11	1.31	1.08	Bal.	138.4		51	Fewer graphite nodules of about 0.060 mm. in dia. Machinability comparable to Example #7. Strength poor but hardness high.

TABLE I. MECHANICAL PROPERTIES AND SUMMARY OF MICROSTRUCTURE FOR VARIOUS Ni-Be-C HEATS
[All material in solution annealed and aged condition (AT) unless otherwise specified]

Example	Chemical Composition in Percent				Tensile Properties UTS in K s.i.	Percent El.	Hardness Rc	Microstructure at 100X, Relative Estimated Machinability and Relative Strength and Hardness
	Be	Mg	C	Ni				
	Alloys with 1.0–1.4% C—Variable Mg Content							
9	2.28	0.40	1.04	Bal	155.6	0.1	48.5	Many graphite nodules about 0.040 mm. in dia. but some irregular graphite shapes. Machinability more than two times that of Example #1. Strength marginal but hardness satisfactory.
10	22.2	0.86	1.22	Bal	198.1	0.5	49.5	Larger graphite nodules of about 0.050 mm. dia. Machinability somewhat better than Example #9. Strength is high and hardness satisfactory.
11	2.13	1.32	1.43	Bal	154.0	0.1	51.5	Larger graphite nodules of about 0.070 mm. dia. and larger irregular graphite shapes. Machinability somewhat better than Example 10. Strength marginal but hardness high.
	Alloys with Low C							
12	1.98	0.45	0.47	Bal	180.0	0.5	51	Small graphite nodules of 0.003 mm. dia. Well dispersed. Machinability better than two times that of Example #1. Strength and hardness satisfactory.
13	20.1	0.61	0.50	Bal	211.8	1.0	51	Graphite nodules of 0.010 mm. in dia. Well dispersed. Machinability somewhat better than Example #12. High strength and hardness.
	Alloys with Low Mg							
14	2.61	0.10	0.60	Bal	175.5	1.2	50	Graphite nodules of 0.010 mm. dia. Well dispersed. Machinability comparable to Example #13. Strength and hardness satisfactory.
	Alloys with Low Be							
15	1.38	0.62	0.59	Bal	168.7	2.0	45.5	Graphite nodules of 0.030 mm. in dia. Well dispersed. Machinability more than two times that of Example #1. Strength and hardness low because of low Be content.
	Alloys with High Be							
16	29.5	0.15	0.83	Bal	192.7	----------	51	Graphite nodules of 0.020 mm. in dia. and well dispersed. Machinability more than two times that of Example #1. High strength and hardness.

Preparation, processing and metallurgical evaluation of Ni-Be-C-Ca-Si and Ni-Be-C-Ce alloys

Heats of Ni-Be-C alloys were made by melting in air, using a 3000-cycle induction furnace of 35-lb. capacity and lined with magnesia. Charges consisted of sheared, electrolytic nicked squares, beryllium pebbles and solid, dry graphite. The nickel was melted and heated to 2750° F. and the melt "killed" with a small addition of beryllium pebbles. The desired amount of graphite was added by stirring until it dissolved into the melt. The remainder of the beryllium pebbles were added and the melt temperature lowered to 2525° F. The Ca and Ce were added in the form of Si–30% Ca master alloy and metallic Ce shavings. These were added and stirred into the heat just before pouring. The melts were poured into phosphate

MICROSTRUCTURE AND HARDNESS OF Ni-Be-C ALLOYS TREATED WITH Si-30% Ca AND Ce ADDITIONS
[All material in solution annealed and aged condition]

Example	Chemical Composition in percent					Rockwell Hardness	Microstructure at 100x. Relative Estimated Machinability and Relative Hardness and Strength
	Be	C	Ca	Si	Ce		
17	0.92	0.64	0.20	0.80	----------	B–92	Graphite nodules of about 0.010 mm. dia. and well distributed. Machinability more than two times that of Example #1. Hardness unsatisfactorily low.
18	2.01	0.64	.025	0.39	----------	C–47	No graphite nodules but irregular shape Machinability comparable to Example #17. Hardness satisfactory but strength would be <150,000 p.s.i.
19	2.17	0.54	0.20	0.89	----------	C–51	Graphite nodules of 0.010 mm. dia. and well dispersed. Machinability more than two times that of Example #1. Hardness satisfactory as would be strength.
20	2.07	0.49	0.52	1.98	----------	C–51	Graphite nodules of about 0.020 mm. dia. and well dispersed. Machinability comparable to Example #19. Hardness satisfactory as would be strength.
21	2.99	0.67	0.26	0.97	----------	C–52	Graphite nodules of about 0.010 mm. dia. and well dispersed. Machinability comparable to Examples 19 and 20. Hardness satisfactory as should be strength.
22	1.22	0.74	----------	----------	0.20	B–84	No graphite present but interdendritically dispersed intermetallic phase. Machinability poorer than two times that of Example #1. Hardness unsatisfactory as would be strength.
23	2.20	0.75	----------	----------	0.10	C–49	Graphite nodules of 0.010 mm. dia. and well dispersed. Machinability better than two times that of commercial alloy Example #1. Hardness satisfactory as would be strength.
24	2.21	0.66	----------	----------	0.12	C–46	Graphite nodules of 0.005 mm. dia. and well dispersed. Machinability and strength comparable to Example #23. Hardness is marginal.
25	2.25	0.78	----------	----------	0.18	C–42	Graphite nodules of 0.010 mm. dia. present but mixed with interdendritic intermetallic phase. Machinability questionable. Hardness and strength unsatisfactorily low.
26	2.18	0.78	----------	----------	0.52	C–41	All graphite in form of irregular shapes. Considerable intermetallic phase present. Machinability questionable. Strength and hardness unsatisfactorily low.
27	3.17	0.75	----------	----------	0.21	C–50	No graphite but considerable intermetallic phase present. Unsatisfactory machinability. Hardness high as would be strength.

bonded sand molds having 1" diameter x 13" long cavities. The castings were annealed and then trimmed from the gates and sprues by sawing and specimens were prepared for micro-structural and hardness evaluation by turning. Specimens were examined in the solution annealed (water quenched from 1950° F.) and aged (3 hours at 950° F.) condition (AT). Qualitative machinability observations were made during the sawing and turning operations while preparing metallurgical evaluation specimens.

From the data in the above tables, it will be seen that Examples 3 through 16 inclusive, 19 through 21 inclusive and 23 and 24 fall into an acceptable commercial strength and/or hardness range with estimated machinable characteristics of at least twice that of the commercial alloy, Example 1. The compositions of these alloys are thus all included in the desired embodiments of the invention.

Preparation, processing and machinability evaluation of typical alloys of the invention

(A) *Melting, casting and heat treatment.*—Heats of nickel-beryllium alloy were made by melting in air sheared squares of electrolytic nickel in a 3000-cycle, 100-kw. induction furnace lined with magnesia of 300-lb. capacity. The nickel was melted at 2750° F., sufficient weight of beryllium pebbles were added to produce the desired beryllium composition and the molten metal temperature lowered to 2500° F. Graphite was added in weighed amounts of dry, solid material stirred into the melt. Then Mg was added as a Ni–13 to 16% Mg master alloy just before pouring and stirred in. The heats were poured at 2525° F. into sand molds having cavities of 3½" diameter x 13" long. The resultant round bars were cut from the sprues and risers and either left "as cast," solution annealed; i.e., heated to 1950° F. for three hours and water quenched, or furnace annealed; i.e., heated to 1950° F. for three hours in a large muffle furnace and then cooled to room temperature with the furnace when its power was cut off.

ANALYSIS AND HARDNESS OF AS-CAST AND ANNEALED NICKEL-BERYLLIUM ALLOYS

Example No.	Composition in Percent				Heat Treated Condition	Hardness	
	Be	C	Mg	Ni		BHN	Rockwell C
28	2.58	.08	.01	Bal.	Furnace Annealed	229	20
29	2.15	.84	.57	Bal.	Solution Annealed	341	37
30	2.11	.45	.38	Bal.	Solution Annealed	321	35
31	2.20	.57	.64	Bal.	As Cast	247	24
32a	2.29	.70	.49	Bal.	Solution Annealed	341	37
32b	2.29	.70	.49	Bal.	Furnace Annealed	229	20

(B) *Machinability tests*:

(1) Turning—

(a) *Machine*—American Pacemaker Lathe with variable speed 30 HP motor.

(b) *Tool material*—Grade C–2 Carbide, specifically Carboloy Grade 883.

(c) *Tool geometry*—

	Degree
Bake rake	0
Side rake	5
Side cutting edge angle	15
End cutting edge angle	15
Relief	5

Nose radius, 1/32".

(d) *Cutting fluid*—Water soluble oil diluted 1:20.

(e) *Tool life end point*—At a wearland of 0.016" or nose breakdown.

(f) *Tool life at*—

0.009 in./rev.—Feed
0.062"—Depth of cut
90 ft./min.—Cutting speed

Example No.:	Tool life in minutes
28	1.5
29	12
30	14
31	3
32a	19
32b	40

Tool life of Example $\frac{32b}{28}$=26.7

(g) Speed at 30 min. tool life (with conditions as in (f) above).

Example No.:	Cutting speed ft./min.
28	46
29	41
30	52
31	62
32a	58
32b	92

Cutting speed of Example $\frac{32b}{28}$=2

(2) Drilling—

(a) *Machine*—Cincinnati 16" Box Column Drill Press with variable speed.

(b) *Tool material*—Molybdenum-type high speed steel.

(c) *Tool geometry*—

Diameter	in.	0.250
Length	in.	2.5
Point angle	deg.	7
Point style	deg.	7
Point grind		Plain
Helix angle	deg.	29

(d) *Cutting fluid*—Water soluble oil diluted 1:20.

(e) Drill life end point—At a 0.016" wearland or breakdown.

(f) Number of holes in ¾" plate at:
40 ft./min.
Cutting speed
0.005"/rev.-feed

Example No.:	No. of holes before drill life end point
28	80
32b	165

No. of holes for Example $\frac{32b}{28}$=2.06

From the above closely controlled machining tests evaluating typical alloys of the invention as well as a commercial alloy (Example 28) in the furnace annealed condition for comparison purposes, it is seen that a typical alloy of the invention, Example 32*b*, can be turned with 26.7 times more tool life at comparable feed, depth of cut and cutting speed than the commercial alloy, Example 28. The typical alloy of the invention can be turned at twice the cutting speed to give similar tool life in turning operations. Twice as many holes can be drilled under identical conditions into a typical alloy of the invention

compared to the commercial alloy before the drill life end point. These closely controlled machinability evaluations show that the alloys of the invention have approximately 2 to 25 times the machinability of commercial alloys, depending upon how the machinability is measured.

Examples 29 through 32*b* compare closely in composition with Example 3 in Table I. Also, the microstructures of these examples were almost identical. This was to be expected because the preparation, melting and casting were very similar.

Further evidence of the improved machinability was clearly demonstrated in uncontrolled operations when the various alloys of the invention were sawed and turned into tensile, hardness and metallographic samples. Whenever graphite was both present and well distributed, a marked increase in machinability over the commercial alloys was readily observed.

Although particular embodiments of the present invention are shown and described for purposes of illustration, it is apparent that changes and modifications may be made without departing from the broader aspects of this invention. The invention described herein is therefore intended to be construed to encompass all modifications thereon within the scope of the appended claims.

Having thus described my invention, I claim:

1. A nickel-base alloy consisting essentially of:
(a) beryllium from about 0.90% to about 3.00%;
(b) carbon from about 0.35% to about 1.30%; and
(c) a sufficient amount of a metal selected from the group consisting of magnesium, calcium and cerium to uniformly disperse the carbon in nodular form.

2. An alloy according to claim 1, wherein:
the magnesium ranges from about 0.1% to about 1.35%; the calcium ranges from about 0.05% to about 0.55%; and the cerium ranges from about 0.10% to about 0.20%.

3. A nickel-base alloy consisting essentially of:
beryllium from about 2.0% to about 2.25%; carbon from about 0.50% to about 0.75%; and from about 0.05% to about 1.35% of a metal selected from the group consisting of magnesium, calcium and cerium.

4. An alloy according to claim 3, wherein:
magnesium ranges from about 0.15% to about 0.50%; calcium ranges from about 0.20% to about 0.30%; and cerium ranges from about 0.10% to about 0.15%.

5. An alloy consisting essentially of:
beryllium from about 2.00% to about 2.25%; carbon from about 0.50% to about 0.75%; magnesium from about 0.15% to about 0.50%; balance nickel.

6. An alloy according to claim 5, wherein:
beryllium ranges from about 2.00% to about 2.25%; carbon ranges from about 0.50% to about 0.75%; calcium ranges from about 0.20% to about 0.30%; balance nickel.

7. An alloy according to claim 5, wherein:
beryllium ranges from about 2.00% to about 2.25%; carbon ranges from about 0.50% to about 0.75%; cerium ranges from about 0.10% to about 0.15%; balance nickel.

8. An alloy according to claim 1, wherein:
a metal selected from the group consisting of misch metal, lanthanum, and neodymium is substituted for the cerium.

9. An alloy according to claim 3, wherein:
a metal selected from the group consisting of misch metal, lanthanum, and neodymium is substituted for the cerium.

10. An alloy according to claim 7, wherein:
a metal selected from the group consisting of misch metal, lanthanum, and neodymium is substituted for the cerium.

11. A method for heat treating a nickel-based alloy, consisting essentially of:
beryllium from about 0.90% to about 3.0%;
carbon from about 0.35% to about 1.30%; and
a sufficient amount of a metal selected from the group consisting of magnesium, calcium and cerium to uniformly disperse the carbon in nodular form, comprising:
heating the alloy at about 1950° F. until the beryllium is in solid solution with the nickel matrix;
quenching the heated alloy;
reheating the quenched alloy at from about 900° F. to about 1000° F. until a predetermined tensile strength is developed; and
cooling the heated alloy to room temperature.

12. The method according to claim 11, wherein:
the alloy is quenched by contact with a material selected from the group consisting of oil, air and water.

13. The method according to claim 11, wherein the selected metal is from about 0.05% to about 1.35%.

14. The method according to claim 1, wherein the alloy is first heated for about three hours at about 1950° F.

15. The method according to claim 11, wherein the alloy is reheated from about one to about three hours at about 900° F. to about 1000° F.

16. A method for heat treating a nickel-based alloy, consisting essentially of:
beryllium from about 2.0% to about 2.25%;
carbon from about 0.5% to about 0.75%; and
from about 0.05% to about 1.35% of a metal selected from the group consisting of magnesium, calcium and cerium.

17. The method according to claim 16, wherein the alloy is first heated for about three hours at about 1950° F.

18. The method according to claim 16, wherein the alloy is reheated from about one to about three hours at about 900° F. to about 1000° F.

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