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(54) **Heat treatment of nickel-base superalloy die inserts**

(57) A rigid die insert for forming and shaping a working material comprises a nickel-base superalloy, preferably Rene 95. A plurality of gamma prime particles are uniformly distributed throughout the rigid die insert, which has a Rockwell hardness Rc of between about 48 and about 52. The invention also includes a method of treating a rigid die insert comprising a nickel-base superalloy to reduce crack propagation and raise yield stress. The method comprises the steps of: providing

the rigid die insert; dissolving larger gamma-prime particles in the rigid die insert; and growing additional gamma-prime particles of smaller particle size in the rigid die insert, whereby the particle size of each of the plurality of gamma-prime particles is refined, thereby reducing crack propagation and raising the yield stress of the rigid die insert. A method of refining the particle size of gamma-prime particles in a Rene 95 superalloy is also disclosed.

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Description

[0001] The present invention relates to die inserts formed from nickel-based superalloys, and, more particularly but not exclusively, to rigid die inserts formed from the superalloy Rene 95 and having improved resistance to crack propagation and yield stress.

[0002] Hot forming operations require rigid dies to form and shape the working material. The dies are exposed to extreme temperatures and pressures and can fail under a number of life-limiting conditions such as excessive wear, cracking or heat-checking. Furthermore, die replacement is costly in terms of material expense, machining effort and operation downtime. Efforts to increase die life are constantly sought. To limit replacement costs, die inserts are formed from materials such as nickel-base superalloys that can withstand the extreme conditions encountered during hot forming. Therefore, what is needed is a rigid die insert, formed from a superalloy, that has improved resistance to crack propagation and improved yield stress. What is also needed is a method of treating rigid die inserts formed from a nickel-base superalloy that will increase the life of the die by improving the crack propagation resistance and yield strength of the die insert.

[0003] The present invention meets these needs and others by providing a rigid die insert having an extended usable life when used in hot forming operations. The rigid die insert is made from a nickel-base superalloy, preferably Rene 95, having fine-grained gamma-prime (γ') particles uniformly distributed throughout the die.

[0004] The invention also provides a heat treatment process for dies formed from a nickel-base superalloy, preferably Rene 95, to extend usable life of the die by improving the crack propagation resistance and yield strength of the die.

[0005] Accordingly, one aspect of the present invention is to provide a rigid die insert for forming and shaping a working material. The rigid die insert comprises a nickel-base superalloy, wherein a plurality of gamma prime particles are uniformly distributed throughout the rigid die insert, and wherein the rigid die insert has a Rockwell hardness, R_C , of between about 48 and about 52.

[0006] A second aspect of the present invention is to provide a nickel-base superalloy for forming a rigid die insert. The nickel-base superalloy comprises a Rene 95 alloy and is formed by heating the Rene 95 alloy to a sub-solvus temperature for a first predetermined hold time, quenching the Rene 95 in a room temperature bath, and heating the Rene 95 alloy to a second predetermined temperature for a second predetermined hold time. The nickel-base superalloy has a plurality of gamma prime particles uniformly distributed throughout, and a Rockwell hardness, R_C , of between about 48 and about 52.

[0007] A third aspect of the present invention is to provide a rigid die insert for forming and shaping a working

material. The rigid die insert comprises a treated Rene 95 superalloy, the Rene 95 superalloy being treated by heating the Rene 95 superalloy to a sub-solvus temperature for a first predetermined hold time, quenching the Rene 95 superalloy in a room temperature bath, and heating the Rene 95 superalloy to a second predetermined temperature for a second predetermined hold time. The treated Rene 95 superalloy has a plurality of gamma prime particles uniformly distributed throughout, and a Rockwell hardness, R_C , of between about 48 and about 52.

[0008] A fourth aspect of the present invention is to provide a method of treating a rigid die insert to reduce crack propagation and raise yield stress, wherein the rigid die insert comprises a nickel-base superalloy having a plurality of gamma-prime particles, each of the gamma-prime particles having a particle size. The method comprises the steps of: providing the rigid die insert; dissolving larger gamma-prime particles in the rigid die insert; and growing additional gamma-prime particles of smaller particle size in the rigid die insert, whereby the particle size of each of the plurality of gamma-prime particles is refined, thereby reducing crack propagation and raising the yield stress of the rigid die insert.

[0009] A fifth aspect of the present invention is to provide a method of refining the particle size of gamma-prime particles in a Rene 95 superalloy. The method comprises the steps of: providing a Rene 95 superalloy; heating the Rene 95 superalloy to a first temperature, the first temperature being a temperature below a solvus temperature of the Rene 95 superalloy thereby dissolving larger gamma-prime particles; quenching the Rene 95 superalloy at room temperature in a bath; and aging the Rene 95 superalloy at a second predetermined temperature for a second predetermined hold time, thereby growing additional gamma-prime particles of smaller particle size, whereby a more uniform size distribution of gamma-prime particles is created.

[0010] A sixth aspect of the present invention is to provide a method of treating a rigid die insert to reduce crack propagation and raise yield stress. The rigid die insert comprises a Rene 95 superalloy having a plurality of gamma-prime particles, each of the gamma-prime particles having a particle size. The method comprises the steps of: providing the rigid die insert; heating the rigid die insert to a first temperature for a first predetermined hold time, the first temperature being a temperature below a solvus temperature of the Rene 95 superalloy; forced-air cooling the rigid die insert; quenching the rigid die insert at room temperature in a bath, thereby dissolving larger gamma-prime particles; and aging the rigid die insert at a second predetermined temperature for a second predetermined hold time, whereby the particle size of each of the plurality of gamma-prime particles is refined, thereby reducing crack propagation and raising the yield stress of the rigid die insert.

[0011] The invention will now be described in greater detail, by way of example, with reference to the draw-

ings, in which:-

FIGURE 1 is a schematic representation of a die assembly having a rigid die insert of the present invention; and

FIGURE 2 is a plot of die lifetime for die inserts of the present invention versus prior-art die inserts.

[0012] In the following description, like reference characters designate like or corresponding parts throughout the several views shown in the figures. It is also understood that terms such as "top," "bottom," "outward," "inward," and the like are words of convenience and are not to be construed as limiting terms.

[0013] Referring to the drawings in general and to Figure 1 in particular, it will be understood that the illustrations are for the purpose of describing a preferred embodiment of the invention and are not intended to limit the invention thereto. Figure 1 is a schematic cross-sectional view of a die assembly with the die insert of the present invention. Die 10, which is used to hot work working material 30, typically comprises two half segments 12 and 22. Each half segment 12 and 22 has an outer shell 14 and rigid die insert 16. The outer shell 14 may be formed from a material such as, but not limited to, high strength tool steel.

[0014] The present invention provides a rigid die insert 16 having an extended usable life when used in hot forming operations. The rigid die insert 16 is made from a nickel-base superalloy, preferably Rene 95, and contains a plurality of fine-grained gamma-prime (γ') particles uniformly distributed throughout the die. Rene 95 is a nickel-base superalloy comprising: between about 12 and 14 weight percent chromium; between about 7 and 9 weight percent cobalt; between about 3.3 and about 3.7 weight percent molybdenum; between about 3.3 and about 3.7 weight percent tungsten; between about 3.3 and about 3.7 weight percent niobium; between about 2.3 and about 2.7 weight percent titanium; between about 3.3 and about 2.7 weight percent aluminum; between about 0.04 and about 0.09 weight percent carbon; between about 0.006 and about 0.016 weight percent boron; between about 0.03 and about 0.07 weight percent zirconium; with the balance of the alloy being nickel. The present invention also provides a heat treatment process for dies made of the superalloy Rene 95 to extend the usable life of the die 10 when it is used in hot forming operations. The heat treatment has been shown to increase die life by over 87%.

[0015] As-received superalloy material, such as Rene 95, typically comprises a combination of large and small gamma prime (γ') particles in a nickel matrix with a Rockwell hardness, R_C , of about 42. The central idea of the heat treatment of the present invention is to inhibit crack propagation and raise the yield stress by eliminating large gamma prime particles and thus obtain a more uniform size distribution of the gamma prime particles. This

is achieved through heating the superalloy material to a sub-solvus temperature, followed by rapid cooling in a room temperature bath. This combination of heating and quenching dissolves the large gamma prime particles and creates a more uniform distribution of fine gamma prime particles. Following the quench, additional fine gamma prime precipitate are grown from solution by an aging process, which includes heating to a predetermined temperature for a specified hold time. Superalloy materials that are heat treated according to the present invention exhibit an increase in hardness. The measured Rockwell hardness, R_C , of Rene 95 material after the heat treatment according to the present invention is in the range from about 48 to about 52.

[0016] The cooling, or quench, rate must be fast enough to lock in the desirable microstructure. At the same time, however, the cooling rate must be sufficiently slow to avoid cracking the material due to thermal gradient-induced stresses. Cracks can be initiated at the surface or center of the material due to the rapidly cooling material that shrinks and yields plastically in tension. These stresses must be kept below the ultimate strength of the material.

[0017] In the present invention, a rigid die insert formed from a nickel-base superalloy, preferably Rene 95, is heated slowly to a sub-solvus temperature; i.e., below the solvus temperature of the particular nickel-base superalloy. For rigid die inserts formed from Rene 95, the rigid die insert 16 is heated to about 2050°F in an inert atmosphere, such as argon, and held at temperature for about 2 hours. Upon removal from oven, the rigid die insert 16 is immediately cooled with forced air provided by a fan or any equivalent device. After a period of air cooling, the duration of which is governed by the before mentioned constraints on yielding, the rigid die insert 16 is quickly quenched in a room-temperature bath. For rigid die inserts of approximately 4"x2"x1½" in size, the air cooling duration is about 30 seconds. An oil bath is preferably used for the quench bath. The rigid die insert 16 is left in the bath until safe removal is possible. Finally, the rigid die insert 16 is aged by slowly heating the rigid die insert 16 to a second temperature for a predetermined hold time. For rigid die inserts 16 formed from Rene 95, the aging step comprises heating the rigid die insert 16 to about 1400°F in an inert atmosphere, such as argon, and holding the rigid die insert 16 at temperature for about 16 hours.

[0018] The most appropriate sub-solvus temperature and cooling rate is dependent on the size and geometry of the rigid die insert 16 that is heat treated. Whereas the heat treatment of the present invention has been optimized for dies of approximately 4"x2"x1½" in size, a similar heat treatment, with minor modifications to cooling rates, is appropriate for larger or smaller dies or dies of differing geometry.

[0019] The following example serves to illustrate the advantages and features of the present invention.

Example 1

[0020] Parts were hot formed using three types of dies: a die of an earlier design having a Rene 95 die insert that was not heat treated according to the present invention; a redesigned die having a Rene 95 die insert that was not heat treated according to the present invention; and a redesigned die having a Rene 95 die insert that was heat treated according to the present invention. The average lives of the different die types, represented by the average number of pieces produced before die failure, are compared in Figure 2. The die of earlier design ("Old Style Die" in Figure 2) produced an average of 975 pieces before failure. The redesigned die which did not have a Rene 95 die insert that was heat treated according to the present invention ("New Style Die" in Figure 2) produced an average of 1442 pieces before failure. Finally, the redesigned die having a Rene 95 die insert that was heat treated according to the present invention ("New Style Die & New Heat Treat" in Figure 2) produced an average of 2696 pieces before failure. The results indicate that the redesigned die inserts that were heat treated according to the present invention alone increased die life by 87% over the redesigned die having Rene 95 die inserts that were not treated according to the present invention.

[0021] While various embodiments are described herein, it will be apparent from the specification that various combinations of elements, variations, or improvements thereon may be made by those skilled in the art, and are thus within the scope of the invention. For example, an entire die may be formed from a nickel-base alloy and heat treated according to the method described herein.

[0022] For the sake of good order, various aspects of the invention are set out in the following clauses:-

1. A rigid die insert for forming and shaping a working material, said rigid die insert comprising a nickel-base superalloy, wherein a plurality of gamma prime particles are uniformly distributed throughout said rigid die insert, and wherein said rigid die insert has a Rockwell hardness, R_C , of between about 48 and about 52.

2. The rigid die insert according to Clause 1, wherein said nickel-base superalloy is Rene 95.

3. A nickel-base superalloy for forming a rigid die insert, said nickel-base superalloy comprising a Rene 95 alloy and being formed by heating said Rene 95 alloy to a sub-solvus temperature in an inert atmosphere for a first predetermined hold time, quenching said Rene 95 in a room temperature bath, and heating said Rene 95 alloy to a second predetermined temperature for a second predetermined hold time in an inert atmosphere, wherein said nickel-base superalloy has a plurality of gamma

prime particles uniformly distributed throughout, and wherein said nickel-base superalloy has a Rockwell hardness, R_C , of between about 48 and about 52.

4. The nickel-base superalloy of Clause 3, wherein said sub-solvus temperature is about 2050°F, and wherein said first predetermined hold time is about two hours.

5. The nickel-base superalloy of Clause 3, wherein said second predetermined temperature is about 1400°F, and wherein said second predetermined hold time is about 16 hours.

6. A rigid die insert for forming and shaping a working material, said rigid die insert comprising a Rene 95 superalloy, wherein said rigid die insert is heated in an inert atmosphere to a sub-solvus temperature of said Rene 95 superalloy for a first predetermined hold time, quenched in a room temperature bath, and heated in an inert atmosphere to a second predetermined temperature for a second predetermined hold time, wherein said rigid die insert has a plurality of gamma prime particles uniformly distributed throughout, and wherein said rigid die insert has a Rockwell hardness, R_C , of between about 48 and about 52.

7. The rigid die insert of Clause 6, wherein said sub-solvus temperature is about 2050°F, and wherein said first predetermined hold time is about two hours.

8. The rigid die insert of Clause 6, wherein said second predetermined temperature is about 1400°F, and wherein said second predetermined hold time is about 16 hours.

9. A method of treating a rigid die insert to reduce crack propagation and raise yield stress therein, the rigid die insert comprising a nickel-base superalloy having a plurality of gamma-prime particles, each of the gamma-prime particles having a particle size, the method comprising the steps of:

providing the rigid die insert;
dissolving larger gamma-prime particles in the rigid die insert; and growing additional gamma-prime particles of smaller particle size in the rigid die insert,
whereby the particle size of each of the plurality of gamma-prime particles is refined, thereby reducing crack propagation and raising the yield stress of the rigid die insert.

10. The method of Clause 9, wherein the step of dissolving larger gamma-prime particles comprises

the steps of:

temperature for a first predetermined hold time,
said first predetermined temperature being a
sub-solvus temperature; and
quenching the rigid die insert in a room temper-
ature bath.

11. The method of Clause 10, further including the
step of forced-air cooling the rigid die insert after the
step of heat treating the rigid die insert to a first pre-
determined temperature.

12. The method of Clause 10, wherein the inert at-
mosphere is an argon atmosphere.

13. The method of Clause 10, wherein the step of
quenching the rigid die insert in a room temperature
bath comprises quenching the rigid die insert in a
room temperature oil bath.

14. The method of Clause 9, wherein the step of
growing additional gamma-prime particles of small-
er particle size comprises aging the rigid die insert
in an inert atmosphere to a second predetermined
temperature for a second predetermined hold time.

15. The method of Clause 14, wherein the inert at-
mosphere is an argon atmosphere.

16. A method of refining the particle size of gamma-
prime particles in a Rene 95 superalloy, the method
comprising the steps of:

providing a Rene 95 superalloy;
heating the Rene 95 superalloy in an inert at-
mosphere to a first temperature, the first tem-
perature being a temperature below a solvus
temperature of the Rene 95 superalloy;
quenching the Rene 95 superalloy at room tem-
perature in a bath, thereby dissolving larger
gamma-prime particles; and
aging the Rene 95 superalloy in an inert atmos-
phere at a second predetermined temperature
for a second predetermined hold time, thereby
growing additional gamma-prime particles of
smaller particle size, whereby a more uniform
size distribution of gamma-prime particles is
created.

17. The method of Clause 16, wherein the step of
heating the Rene 95 superalloy in an inert atmos-
phere to a first temperature comprises heating the
Rene 95 superalloy to about 2050°F for about two
hours.

18. The method of Clause 16, wherein the step of
quenching the Rene 95 superalloy at room temper-

ature in a bath comprises quenching the Rene 95
superalloy in a room temperature oil bath.

19. The method of Clause 16, wherein the step of
aging the Rene 95 superalloy in an inert atmos-
phere at a second predetermined temperature for a
second predetermined hold time comprises heating
the Rene 95 into about 1400°F for about 16 hours.

20. The method of Clause 16, wherein the inert at-
mosphere is an argon atmosphere.

21. A method of treating a rigid die insert to reduce
crack propagation and raise yield stress, the rigid
die insert comprising a Rene 95 superalloy having
a plurality of gamma-prime particles, each of the
gamma-prime particles having a particle size, the
method comprising the steps of:

providing the rigid die insert;
heating the rigid die insert in an inert atmos-
phere to a first temperature for a first predeter-
mined hold time, the first temperature being a
temperature below a solvus temperature of the
Rene 95 superalloy;
forced-air cooling the rigid die insert;
quenching the rigid die insert at room temper-
ature in a bath, thereby dissolving larger gam-
ma-prime particles; and
aging the rigid die insert in an inert atmosphere
at a second predetermined temperature for a
second predetermined hold time,
whereby the particle size of each of the plurality
of gamma-prime particles is refined, thereby re-
ducing crack propagation and raising the yield
stress of the rigid die insert.

22. The method of Clause 21, wherein the step of
quenching the rigid die insert in a room temperature
bath comprises quenching the rigid die insert in a
room temperature oil bath.

23. The method of Clause 21, wherein the step of
heating the rigid die insert in an inert atmosphere to
a first temperature for a first predetermined hold
time comprises heating the rigid die insert to about
2050°F for about two hours.

24. The method of Clause 21, wherein the step of
aging the rigid die insert in an inert atmosphere at
a second predetermined temperature for a second
predetermined hold time comprises heating the rig-
id die insert into about 1400°F for about 16 hours.

25. The method of Clause 21, wherein the inert at-
mosphere is an argon atmosphere.

Claims

1. A rigid die insert for forming and shaping a working material, said rigid die insert comprising a nickel-base superalloy, wherein a plurality of gamma prime particles are uniformly distributed throughout said rigid die insert, and wherein said rigid die insert has a Rockwell hardness, R_C , of between about 48 and about 52. 5
2. The rigid die insert according to Claim 1, wherein said nickel-base superalloy is Rene 95. 10
3. A nickel-base superalloy for forming a rigid die insert, said nickel-base superalloy comprising a Rene 95 alloy and being formed by heating said Rene 95 alloy to a sub-solvus temperature in an inert atmosphere for a first predetermined hold time, quenching said Rene 95 in a room temperature bath, and heating said Rene 95 alloy to a second predetermined temperature for a second predetermined hold time in an inert atmosphere, wherein said nickel-base superalloy has a plurality of gamma prime particles uniformly distributed throughout, and wherein said nickel-base superalloy has a Rockwell hardness, R_C , of between about 48 and about 52. 15 20 25
4. The nickel-base superalloy of Claim 3, wherein said sub-solvus temperature is about 2050°F, and wherein said first predetermined hold time is about two hours. 30
5. A rigid die insert for forming and shaping a working material, said rigid die insert comprising a Rene 95 superalloy, wherein said rigid die insert is heated in an inert atmosphere to a sub-solvus temperature of said Rene 95 superalloy for a first predetermined hold time, quenched in a room temperature bath, and heated in an inert atmosphere to a second predetermined temperature for a second predetermined hold time, wherein said rigid die insert has a plurality of gamma prime particles uniformly distributed throughout, and wherein said rigid die insert has a Rockwell hardness, R_C , of between about 48 and about 52. 35 40 45
6. The rigid die insert of Claim 5, wherein said sub-solvus temperature is about 2050°F, and wherein said first predetermined hold time is about two hours. 50
7. A method of treating a rigid die insert to reduce crack propagation and raise yield stress therein, the rigid die insert comprising a nickel-base superalloy having a plurality of gamma-prime particles, each of the gamma-prime particles having a particle size, the method comprising the steps of: 55
 - providing the rigid die insert;
 - dissolving larger gamma-prime particles in the rigid die insert; and growing additional gamma-prime particles of smaller particle size in the rigid die insert,
 - whereby the particle size of each of the plurality of gamma-prime particles is refined, thereby reducing crack propagation and raising the yield stress of the rigid die insert.
8. The method of Claim 7, wherein the step of dissolving larger gamma-prime particles comprises the steps of:
 - temperature for a first predetermined hold time, said first predetermined temperature being a sub-solvus temperature; and
 - quenching the rigid die insert in a room temperature bath.
9. A method of refining the particle size of gamma-prime particles in a Rene 95 superalloy, the method comprising the steps of:
 - providing a Rene 95 superalloy;
 - heating the Rene 95 superalloy in an inert atmosphere to a first temperature, the first temperature being a temperature below a solvus temperature of the Rene 95 superalloy;
 - quenching the Rene 95 superalloy at room temperature in a bath, thereby dissolving larger gamma-prime particles; and
 - aging the Rene 95 superalloy in an inert atmosphere at a second predetermined temperature for a second predetermined hold time, thereby growing additional gamma-prime particles of smaller particle size, whereby a more uniform size distribution of gamma-prime particles is created.
10. A method of treating a rigid die insert to reduce crack propagation and raise yield stress, the rigid die insert comprising a Rene 95 superalloy having a plurality of gamma-prime particles, each of the gamma-prime particles having a particle size, the method comprising the steps of:
 - providing the rigid die insert;
 - heating the rigid die insert in an inert atmosphere to a first temperature for a first predetermined hold time, the first temperature being a temperature below a solvus temperature of the Rene 95 superalloy;
 - forced-air cooling the rigid die insert;
 - quenching the rigid die insert at room temperature in a bath, thereby dissolving larger gamma-prime particles; and
 - aging the rigid die insert in an inert atmosphere

at a second predetermined temperature for a
second predetermined hold time,
whereby the particle size of each of the plurality
of gamma-prime particles is refined, thereby re-
ducing crack propagation and raising the yield
stress of the rigid die insert. 5

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FIGURE 1

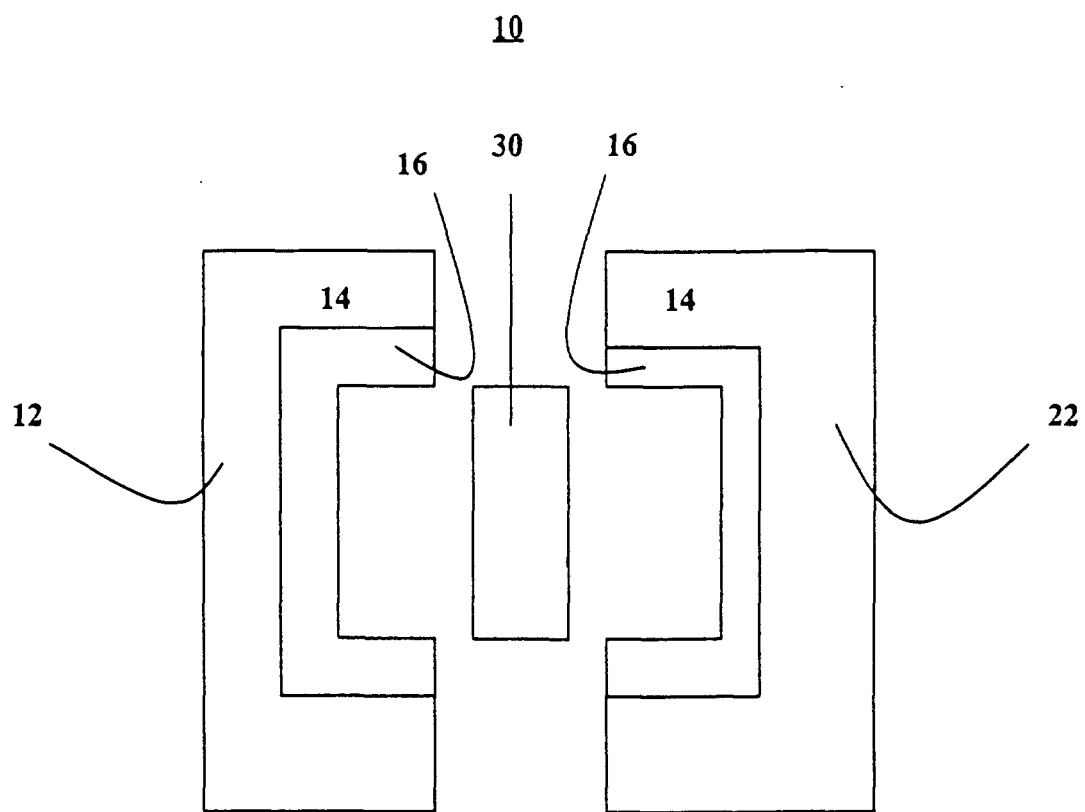


FIGURE 2

