ABSTRACT

A homogeneous and porous abradable seal material structure comprising principally $\gamma$, $\gamma'$ and/or $\beta$ phases for use in elevated temperature operating apparatus consisting essentially of, by weight, 60-80% Ni, 2-12% Cr, 1-10% Co, 4-20% Al, up to 3% of a refractory metal selected from the group consisting of yttrium, hafnium and lanthanum and 3-15% inert powder material selected from the group consisting of diatomaceous earth (D.E.), boron nitride, silicon glass, mica, vermiculite asbestos, molybdenum disulfide, graphite, cobalt oxide, cerium oxide and zinc oxide.

6 Claims, No Drawings
NICKLE BASE HIGH TEMPERATURE ABRADABLE MATERIAL

This is a continuation-in-part of U.S. Pat. application Ser. No. 199,023 filed Nov. 15, 1971, now abandoned.

BACKGROUND OF THE INVENTION

The present invention relates to abradable materials and more particularly relates to a low friction abradable material which is resistant to oxidation at elevated temperatures and is especially suitable for gas turbine engines.

It is known that the efficiency of a gas turbine engine is dependent in part upon the control of gas leakage between stages in both the compressor and turbine sections of the engine. Although the engine is typically designed and manufactured to very precise dimensional tolerances, it is necessary to provide a sufficient cold clearance between the tips of the rotating elements and the surrounding stator assembly to accommodate the differential thermal growth between the parts as the engine assumes its normal operating temperature. To this end, it is necessary to provide the usual manufacturing tolerances plus an additional safety factor to provide for potential engine operation at temperatures in excess of the design temperatures. The requisite clearances thus provided are, however, generally not sufficiently close to permit the engine to operate at its maximum theoretical efficiency.

In an effort to remedy this condition, it has been proposed to utilize an abradable surface on the assembly surrounding the rotating elements and to permit the knife-edge or squeezer tips of the rotor system to penetrate into the coating as a result of thermal expansion, thereby permitting the rotor to seat itself against the casing assembly with what is essentially a zero clearance. A typical abradable seal construction of this type is shown in the U.S. Pat. to Emanuelson et al No. 117,334,131,136 and in copending U.S. Pat. application Ser. No. 161,946 (Attorney's Docket EH-3588) filed July 9, 1971, now abandoned by the present inventors, both being of common assignee with the present invention.

While in theory abradable type seals may be seen to have great potential in improving engine performance, current techniques and abradable seal structures have not been entirely satisfactory in their practical application to current high performance jet engines. In particular, the requirement for seal material which has a high thermal stability and melting point, a relatively constant degree of abradability and good thermal shock characteristics while possessing strong adherence to the metal substrate to which it is applied as well as good structural integrity in an elevated temperature environment up to 2,000°F has not previously been provided.

SUMMARY OF THE INVENTION

The present invention relates to an abradable seal facing material for use in elevated temperature coating apparatus and more particularly relates to a homogeneous and porous nickel-base abradable seal material structure comprising principally γ', γ and β phases for sustained use at temperatures of 1,800°F and up to 2,000°F for short term operation. The present invention also relates to a method for making such a material.

In brief, the present invention contemplates a homogeneous and porous abradable seal material structure comprising principally γ', γ and β phases for use in elevated temperatures operating apparatus consisting essentially of, by weight, 60-80% Ni, 2-12% Cr, 1-10% Co, 4-20% Al, up to 3% of a refractory metal selected from the group consisting of Y, Hf and La and 3-15% inert powder material selected from the group consisting of diatomaceous earth (D.E.), boron nitride, silicon glass, mica, vermiculite asbestos, molybdenum disulfide, graphite, cobalt oxide, cerium oxide and zinc oxide. Preferably, the composition consists essentially of, by weight, 65-75% Ni, 3-9% Cr, 4-8% Co, 7-18% Al, up to 1% Y, Hf or La and 5-10% inert powder material. An optimum composition for such an abradable seal is approximately, by weight, 70-90% Ni, 4.4% Cr, 6.0% Co, 10.4% Al, 0.10% Y and 8.3% diatomaceous earth powder.

Investigations have shown that the total porosity should be established at approximately 35-65%, preferably at approximately 40-60% and most preferably at approximately 50%.

The present invention not only contemplates an abradable seal product but also the process for making the same and more particularly encompasses a method wherein alloy powders selected from the group consisting of Ni-Cr-Fe, Ni-Cr and Co-Al and Co-Cr and Ni-Al are mixed with an inert powder or metal coated inert powder selected from the group consisting of diatomaceous earth, boron nitride, silicon glass, mica, vermiculite asbestos, molybdenum disulfide, graphite, cobalt oxide, cerium oxide and zinc oxide coated with Ni, Co, Cr, Al or alloys thereof and sintered to produce a homogeneous and porous abradable material consisting essentially of, by weight, 60-80% Ni, 2-12% Cr, 1-10% Co, 4-20% Al and 3-15% inert powder. It is preferred to include small amounts of refractory metal selected from the Group 3b or 4b elements, preferably from the group consisting of yttrium, hafnium and lanthanum in order to retard oxide spallation.

In the preferred method, the abradable coating is produced by blending a powder mixture of, by weight, 10-40% nickel-chromium alloy, 5-20% cobalt-aluminum-yttrium alloy and 35-65% coated inert material selected from the group consisting of diatomaceous earth, boron nitride, silicon glass, mica, vermiculite asbestos, molybdenum disulfide, graphite, cobalt oxide, cerium oxide and zinc oxide, said inert material being coated with nickel, cobalt, chromium, aluminum or alloys thereof, and sintering. In a more preferred method, a powder mixture consisting essentially of, by weight, 25-35% nickel-chromium alloy, 12-20% cobalt-aluminum-yttrium alloy and 45-60% nickel or nickel-chromium coated diatomaceous earth material is blended and sintered. The most preferred technique requires the blending and sintering of a powder mixture consisting essentially of, by weight, 30% nickel-chromium alloy, 15% cobalt-aluminum-yttrium alloy and 55% nickel or nickel-chromium coated diatomaceous earth material.

Seals of the above composition have been found to possess unique characteristics which are superior to the prior art abradables as outlined above. In addition, the seals of the present invention have a low thermal conductivity and act as insulators to allow the maintenance of a steep thermal gradient between the hot gas path and the outer diameter of the seal wall while minimizing thermal losses.
DESCRIPTION OF THE PREFERRED EMBODIMENT

The seal material of the present invention is preferably used in conjunction with a holding member such as a conventional metal honeycomb of suitable material and configuration. A variety of metals may be used depending on the specific requirements of the engine. For example, stainless steel such as A.I.S.I. type 321 and nickel or nickel-cobalt base alloys may be employed satisfactorily. As will be appreciated, of course, prior to usage the holding member must be cleaned and degreased by suitable means such as an alkali cleaner or conventional solvents.

After cleaning, alloy powders selected from the group consisting of NiCr, NiAl, NiCr and CoAl, and CoCr and NiAl are thoroughly dry blended and mixed together with inert metal powder selected from the group consisting of diatomaceous earth, boron nitride, silicon glass, mica, vermiculite asbestos, molybdenum disulfide, graphite, cobalt oxide, cerium oxide and zinc oxide and preferably having a coating selected from the group consisting of nickel, cobalt, chromium and aluminum and alloys thereof. The total composition of the powders is selected so as to correspond to, by weight, 60-80% Ni, 2-12% Cr, 1-10% Co, 4-20% Al and 3-15% inert material. The dry powders are mixed with a suitable binder such as a cellulose nitrate solution and packed, as by troweling, into the honeycomb. The carrier is allowed to evaporate and the mixture is then sintered in a nonoxidizing atmosphere, such as argon or a vacuum, according to a schedule selected so as to limit the amount of liquid phase present at any given time to an amount below which will cause the material to slump and thus lose its porosity. As will be appreciated, a satisfactory sintering time-temperature cycle can be varied and depends on the particular composition of the material being treated and its intended application.

The resulting product is a homogeneous abradable seal material comprising principally y, y' and b phases and having a total porosity of approximately 35-65%.

In order further to improve the properties of the above system, a refractory metal powder is preferably added in an amount of up to 3% by weight. Refractory metals selected from the Group 3b and 4b elements such as yttrium, hafnium and lanthanum are satisfactory. The refractory metals should be uniformly distributed in the seal material and are preferably present in an amount of 0.01 to 1.00%, by weight. The refractory metals have been found to increase the adherence of oxide layers such as Al2O3, CoAl2O4, and NiAl2O4 to the seal particles.

It is to be noted that the inert materials serve primarily to increase lubricity and provide an adjustable porosity and density. The inert materials may have a particle size generally within the range of 100 to 200 mesh.

It will be appreciated that the sintering step is executed in order to form a bond between the particles themselves and between the particles and the holding member, as well as to form oxidation resistant alloys. The aluminum containing starting alloy powder, whether it be CoAl, NiAl or CoAlY, NiAlY, etc., acts as an active ingredient. With its inclusion in the pack, there results a liquid phase sintering wherein the sintering process is accelerated with resulting better diffusion and bonding than heretofore experienced.

The abradable seal filler material is in a sense a free standing sinter and is therefore quite porous, the porosity being in the range of approximately 35-65%. It is also in a broad sense a cermet since there is present both metal and oxide which contributes to the structural and physical characteristics of the system.

Particle sizes of the components for the powder mix play an important role in providing a satisfactory seal product in both controlling green density and in controlling the sintering kinetics of the materials system. If the particles are too small, a too dense material is achieved which causes excessive blade wear and if too large, the structural strength of the seal is lessened, the number of bonds per unit volume becomes inadequate and erosion resistance is diminished. The following table sets forth the particle size distributions which have been found most satisfactory:

<table>
<thead>
<tr>
<th>Component</th>
<th>ASTM Sieve</th>
<th>% by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>NiCr</td>
<td>+170</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>-230</td>
<td>5</td>
</tr>
<tr>
<td>NiAl</td>
<td>+170</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>-270</td>
<td>100</td>
</tr>
<tr>
<td>CoAlY</td>
<td>+325</td>
<td>5</td>
</tr>
<tr>
<td>Ni or NiCr</td>
<td>+150</td>
<td>5</td>
</tr>
<tr>
<td>Coated D.E.</td>
<td>-325</td>
<td>30</td>
</tr>
</tbody>
</table>

Experiments were performed varying the relative proportions of coated diatomaceous earth and NiCr to optimize the composition. It was found that with NiD.E. or NiCr/D.E. being varied from 30-85% of the total seal composition, an increase in the amount of coated D.E. resulted in a softening of the sinter while a decrease caused a hardening thereof. Experiments also showed that increasing amounts of NiCr resulted in increasing hardness values for the sinter while decreasing amounts thereof caused a concomitant decrease in hardness. The various ranges expressed hereinbefore as being satisfactory were selected based on dynamic blade tip interactions and erosion testing to match blade configurations and gas velocities in the turbine environment. Other experiments conducted using coated boron nitride, silica glass (Eccospheres™), mica and graphite gave results which were satisfactory, although inferior to the coated diatomaceous earth. Other inert, relatively soft materials which are stable and fairly lubricious, such as cobalt oxide, cerium oxide, zinc oxide, molybdenum disulfide or vermiculite asbestos or the like, may be used. Suitable coatings for the inert powder material are nickel, cobalt, chromium, aluminum or iron or alloys thereof such as nickel-aluminum or nickel-chrome-aluminum.

The abradable seal material of the present invention has shown itself to be suitable for use at sustained operating temperatures up to 1,800°F and able to withstand temperatures up to 2,000°F for short term operation. The composition is resistant to galling, is easily abraded and the utilization of the coated inert diatomaceous earth particles provides insulating and thermal stability characteristics.

In order that those skilled in the art will better understand how the abradable seal of the present invention may be obtained, the following specific examples are
provided. All percentages are by weight unless otherwise noted.

**EXAMPLE I**

A powder mixture having the following composition was thoroughly dry blended and mixed together:

- 30% NiCr (80% Ni, 20% Cr) Metco 43 F NS
- 15% CoAlY (30% Co, 69% Al, 1% Y) - 325 mesh
- 55% Ni-coated D.E. (85% Ni, 15% D.E.)

The above mixture was mixed with a cellulose nitrate solution as a carrier and packed into a Nicraloy™ reinforcing honeycomb foil. After carrier vaporization, the material was sintered in argon at 2,140°F for two hours. The resulting product was a porous homogeneous abradable structure having an open porosity of approximately 40% and a total porosity of approximately 50% with a mean pore size of about 0.001 inch. The structure consisted essentially of γ, γ’, and β phases and had a composition consisting essentially of 70.7% Ni, 6.0% Cr, 4.5% Co, 10.3% Al, 0.2% Y and 8.3% diatomaceous earth.

The sintered seal composition of this example had a density of 2.6 grams/cm³ and exhibited a mean coefficient of thermal expansion from 6.7 × 10⁻⁶ in./in. °F at room temperature to 10.2 × 10⁻⁶ in./in. °F at 1,832°F. After accumulating approximately 600 hours of engine testing, the seal composition remained in good condition with minimal erosion and no spalling from the substrate. In all, it was found that the use of a honeycomb filled with the abradable seal material of the present example, as compared to an unfilled honeycomb, increased the life of the honeycomb by a factor of at least 3. Further, primarily because of its excellent insulating properties, the shroud of the gas turbine engine is rendered more dimensionally stable and is thereby benefited.

**EXAMPLE II**

The techniques of Example I were duplicated on a powder mixture having the following composition:

- 25% NiCr (80% Ni, 20% Cr) Metco 43 F NS
- 20% CoAlY (30% Co, 69% Al, 1% Y) - 325 mesh
- 4% Al flake
- 51% Ni-coated D.E. (85% Ni, 15% D.E.)

After carrier vaporization, the material was sintered in argon at 2,140°F for two hours. The resulting product was a porous homogeneous abradable structure consisting principally of γ, γ’, and β phases of a composition consisting essentially of 63.5% Ni, 5.0% Cr, 6.0% Co, 17.8% Al, 0.2% Y and 7.5% diatomaceous earth and having properties similar to those set forth in Example I.

**Example III**

The techniques of Example I were again duplicated on a powder mixture having the following composition:

- 25% NiCr (80% Ni, 20% Cr) Metco 43 F NS
- 20% CoAlY (30% Co, 69% Al, 1% Y) - 325 mesh
- 55% Ni-coated D.E. (85% Ni, 15% D.E.)

After carrier vaporization, the material was sintered in argon at 1,850°F for two hours and 1,950°F for three hours. The resulting product was a porous homogeneous abradable structure consisting essentially of 66.7% Ni, 5.0% Cr, 6.0% Co, 13.8% Al, 0.2% Y and 8.3% diatomaceous earth and having properties similar to those set forth in Example I.

**Example IV**

The techniques of Example I were again duplicated on a powder mixture having the following composition:

- 25% NiCr (80% Ni, 20% Cr) Metco 43 F NS
- 25% CoAlY (30% Co, 69% Al, 1% Y) - 325 mesh
- 50% Ni-coated D.E. (85% Ni, 15% D.E.)

After carrier vaporization, the material was sintered in argon at 1,850°F for two hours and 1,950°F for three hours. The product was similar to those above and consisted essentially of 62.5% Ni, 5.0% Cr, 7.5% Co, 17.2% Al, 0.3% Y and 7.5% diatomaceous earth.

**Example V**

The techniques of Example I were duplicated on a powder mixture of:

- 35% NiCr (80% Ni, 20% Cr) Metco 43 F NS
- 10% CoAlY (30% Co, 69% Al, 1% Y) - 325 mesh
- 55% Ni-coated D.E. (85% Ni, 15% D.E.)

After carrier vaporization, the material was sintered in argon at 1,750°F for two hours. 1,950°F for two hours and 2,050°F for two hours. The product was similar to those above and consisted essentially of 74.7% Ni, 7.0% Cr, 3.0% Co, 6.9% Al, 0.1% Y and 8.3% diatomaceous earth.

It is to be understood that various modifications may be made without departing from the spirit of the present invention. It is recognized, for example, that while the inert powder material is preferably coated with nickel or an alloy thereof; it may also be coated with cobalt, iron, chromium or aluminum or their alloys.

The invention contemplates the use of superalloy powders with a liquid phase sintering and inert particles coated with a metal, as described.

What has been set forth above is intended primarily as exemplary to enable those skilled in the art in the practice of the invention and it should therefor be understood that within the scope of the appended claims, the invention may be practiced in other ways than as specifically described.

What is claimed is:

1. A homogeneous and porous abradable seal structure consisting principally of γ, γ’, and β phases for use in elevated temperature apparatus consisting essentially of, by weight, 60-80% Ni, 2-12% Cr, 1-10% Co, 4-20% Al, up to 3% of a refractory metal selected from the group consisting of yttrium, hafnium and lanthanum and 3-15% inert powder material selected from the group consisting of diatomaceous earth, boron nitride, silicon glass, mica, vermiculite asbestos, molybdenum disulfide, graphite, cobalt oxide, cerium oxide and zinc oxide.

2. The invention of claim 1 wherein said structure has a total porosity of approximately 35-65%.

3. A homogeneous and porous abradable seal structure consisting principally of γ, γ’, and β phases for use in elevated temperature operating apparatus consisting essentially of, by weight, 65-75% Ni, 3-9% Cr, 4-8% Co, 7-18% Al, up to 1.0% of a refractory metal selected from the group consisting of yttrium, hafnium and lanthanum and 5-10% inert powder material selected from the group consisting of diatomaceous earth, boron nitride, silicon glass, mica, vermiculite asbestos, molybdenum disulfide, graphite, cobalt oxide, cerium oxide and zinc oxide.

4. The invention of claim 3 wherein said structure has a total porosity of approximately 35-65%.
5. A homogeneous and porous abradable seal material structure consisting principally of \( \gamma, \gamma' \) and \( \beta \) phases for use in elevated temperature apparatus consisting essentially of approximately, by weight, 70.9% Ni, 4.4% Cr, 6.0% Co, 10.4% Al, 0.10% Y and 8.3% diatomaceous earth.

6. The invention of claim 5 wherein said structure has a total porosity of approximately 35–65%.