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(54) **Method of hardening a beta titanium member**

(57) A method of hardening the surface of a beta titanium member 11 comprises the step of heating the beta titanium member 11 in a gas mixture consisting essentially of an inert gas and oxygen.

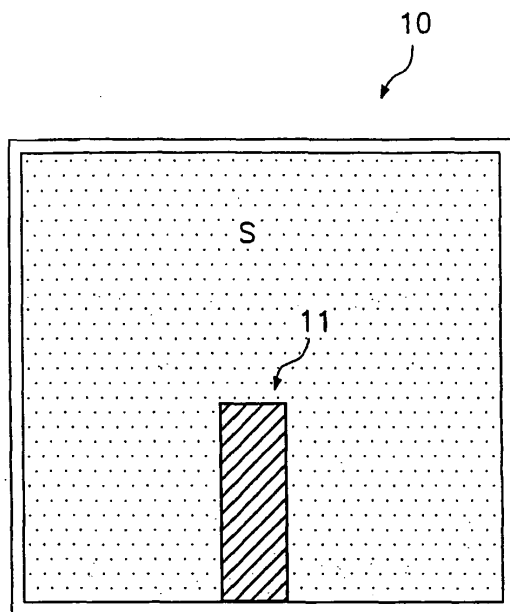


Fig. 1

Description**BACKGROUND OF THE INVENTION**

[0001] The present invention is directed to metal hardening processes and, more particularly, to a method of hardening a beta titanium member.

[0002] In recent years, products made of titanium or of titanium alloy, both of which are lightweight and hard, have become widely used. However, titanium and titanium alloy are active metals and have low wear resistance. Also, surface processing of either material is extremely difficult.

[0003] To overcome such problems, methods have been employed to increase the surface hardness of members formed from such metals. Such methods include forming an outer hardened layer via surface plating or hardening the product surface itself via nitriding or carburizing. However, plating processes encounter the problems of poor adhesion between the plating layer and the titanium surface and damage to the appearance of the titanium, and surface hardening via nitriding or carburizing encounter the problems of coarsening of the product surface and extended processing times.

[0004] Japanese published patent application nos. 2003-73796, 2002-97914 and 2001-81544 disclose further surface hardening methods that employ oxygen diffusion to increase the wear resistance of titanium products. For example, JP 2003-73796 discloses a surface hardening method wherein a titanium member is heated while buried in a highly oxygen-absorbent powder. The powder reduces the oxygen concentration of the atmosphere surrounding the titanium member by physically preventing the titanium surface from coming into contact with oxygen. As a result, a TiO oxygen diffusion layer is formed in the surface of the titanium member while minimizing the formation of an oxidized outer surface layer.

[0005] Although the surface hardness can be increased using such methods, because the titanium member must be buried in oxygen-absorbing powder each time processing is carried out, the process is relatively inefficient and costly. Furthermore, because the titanium member is buried in the oxygen-absorbing powder, the desired cooling rate cannot be obtained following the heat processing, so an appropriate aging treatment cannot be performed.

SUMMARY OF THE INVENTION

[0006] The present invention is directed to various features of a method of hardening a beta titanium member. In one embodiment, a method of hardening the surface of a beta titanium member comprises the step of heating the beta titanium member in a gas mixture consisting essentially of an inert gas and oxygen. Additional inventive features will become apparent from the description below, and such features alone or in combination with the above features may form the basis of further

inventions as recited in the claims and their equivalents.

BRIEF DESCRIPTION OF THE DRAWINGS

[0007]

Fig. 1 shows the basic construction of a particular embodiment of an apparatus for surface hardening a beta titanium member;

Figs. 2A and 2B are graphs of surface hardness for various heat treating methods;

Fig. 3 is a bar graph of the results of friction testing beta titanium members when subjected to the methods shown in figs. 2A and 2B; and

Fig. 4 is a cross sectional diagram of a surface hardened beta titanium member formed according to the methods taught herein.

DETAILED DESCRIPTION OF THE EMBODIMENTS

[0008] Fig. 1 shows the basic construction of a particular embodiment of a beta titanium surface hardening apparatus 10 in the form of a titanium melting furnace for surface hardening a beta titanium member 11. In general, beta titanium member 11 is placed in a processing chamber S of beta titanium surface hardening apparatus 10, and then beta titanium member 11 is heated in an atmosphere comprising a gas mixture comprising oxygen and an inert gas such as argon gas. As a result, heat processing can be carried out in an atmosphere having a lower oxygen concentration than ordinary atmospheric air. In this embodiment, the oxygen concentration ranges from approximately 0.05 vol% to approximately 20 vol% (preferably approximately 1.0 vol% to approximately 10 vol%), the heating temperature ranges from approximately 700°C to approximately 1000°C (preferably approximately 850°C to approximately 950°C), and the heat processing time ranges from approximately 10 minutes to approximately 30 minutes (preferably approximately 15 minutes to approximately 25 minutes).

[0009] After this processing, titanium member 11 undergoes an aging treatment at an ambient temperature of from approximately 400°C to approximately 550°C (preferably approximately 850°C to approximately 950°C) for a time of from approximately 6 hours to approximately 16 hours (preferably approximately 10 hours to approximately 14 hours).

[0010] Figs. 2A and 2B are graphs of surface hardness for various heat treating methods. In Fig. 2A, one line represents an unprocessed beta titanium member, another line represents a beta titanium member subjected to an Argon-Oxygen atmosphere of 5 vol% oxygen at 850°C for 10 minutes, and another line represents a beta titanium member subjected to an Argon-Oxygen atmosphere of 10 vol% oxygen at 850°C for 10 minutes. In Fig. 2B, one line represents an unprocessed beta titanium member, another line represents a beta titanium

member subjected to an Argon-Oxygen atmosphere of 1.7 vol% oxygen at 900°C for 10 minutes, another line represents a beta titanium member subjected to an Argon-Oxygen atmosphere of 5 vol% oxygen at 900°C for 10 minutes, and another line represents a beta titanium member subjected to an Argon-Oxygen atmosphere of 10 vol% oxygen at 900°C for 10 minutes.

[0011] As shown in Fig. 2A, a beta titanium member that was processed at a temperature of 850°C for 10 minutes in an atmosphere having an oxygen concentration of 5 vol% exhibited an HV hardness of 570-400 down to a depth of 0.10 mm (100 µm) below the surface, as compared to the more or less fixed HV hardness of 400 for an unprocessed beta titanium member. In particular, the HV hardness increased to 570-400 from the surface down to a depth of 0.05 mm (50 µm) below the surface. A beta titanium member that was processed at a temperature of 850°C for 10 minutes in an atmosphere having an oxygen concentration of 10 vol% also exhibited an HV hardness of 570-400 down to a depth of 0.10 mm (100 µm) below the surface. In particular, the HV hardness increased to 570-450 from the surface down to a depth of 0.05 mm (50 µm) below the surface.

[0012] As shown in Fig. 2B, a beta titanium member that was processed at a temperature of 900°C for 10 minutes in an atmosphere having an oxygen concentration of 1.7 vol% exhibited an HV hardness of 590-420 from the surface down to a depth of 0.10 mm (100 µm) below the surface, as compared to the more or less fixed HV hardness of 450 for an unprocessed beta titanium member. In particular, the HV hardness increased to 590-495 from the surface down to a depth of 0.05 mm (50 µm) below the surface. A beta titanium member that was processed at a temperature of 900°C for 10 minutes in an atmosphere having an oxygen concentration of 5 vol% exhibited an HV hardness of 580-470 from the surface down to a depth of 0.10 mm (100 µm) below the surface. In particular, the HV hardness increased to 585-515 from the surface down to a depth of 0.05 mm (50 µm) from the surface. A beta titanium member that was processed at a temperature of 900°C for 10 minutes in an atmosphere having an oxygen concentration of 10 vol% exhibited an HV hardness of 545-395 down to a depth of 0.10 mm (100 µm) from the surface. In particular, the HV hardness increased to 545-490 from the surface down to a depth of 0.05 mm (50 µm) below the surface.

[0013] It should be readily apparent from the graphs in Figs. 2A and 2B that, with respect to the temperature parameter, a temperature of 900°C resulted in a greater increase in hardness over a greater range than a temperature of 850°C. More specifically, when the beta titanium member was subjected to a processing temperature of 900°C, the HV hardness declined more gradually beyond a depth of 0.02 mm (20 µm) below the surface than it did when the beta titanium member was subjected to a processing temperature of 800°C. Therefore, taking into consideration the melting temperature of be-

ta titanium, it is preferable that processing be carried out at a temperature in the range of from approximately 850°C to approximately 950°C.

[0014] With respect to the oxygen concentration parameter, Fig. 2B shows that HV hardness increases to a greater degree when the oxygen concentration is 1.7 vol% than when it is 5 vol%. The same is true when the oxygen concentration is 5 vol% than when it is 10 vol%. Therefore, in order to minimize the formation of an oxidized layer while increasing HV hardness, it is preferable that processing be carried out within an oxygen concentration in a range of from approximately 1 vol% to approximately 10 vol%.

[0015] Fig. 3 is a bar graph of the results of friction testing beta titanium members when subjected to the methods shown in Figs. 2A and 2B. The beta titanium member that was heated at 850°C for 10 minutes in an oxygen concentration of 5 vol% is referred to as a first sample, the beta titanium member that was heated at 900°C for 10 minutes in an oxygen concentration of 10 vol% is referred to as a second sample, a beta titanium member that was heated at 900°C for 10 minutes in an oxygen concentration of 5 vol% is referred to as a third sample, and a beta titanium member that was heated at 900°C for 10 minutes in an oxygen concentration of 1.7 vol% is referred to as a fourth sample.

[0016] As shown in Fig. 3, the average amount of wear was 0.15 mm for the unprocessed beta titanium member, 0.138 mm for the first sample, 0.132 mm for the second sample, 0.110 mm for the third sample, and 0.104 mm for the fourth sample. Clearly, the average wear amount was lower for the processed beta titanium members than for the unprocessed beta titanium member. The average wear amount for the third and fourth samples in particular, which were processed at 900°C for 10 minutes, was approximately 30% lower than the wear amount for the unprocessed beta titanium member. Thus, processing at a temperature in a range of from approximately 850°C to approximately 900°C results in wear resistance and surface hardness values that are higher than the equivalent values for an unprocessed beta titanium member.

[0017] From a comparison between the first sample and the third sample, it may be seen that the average amount of wear can be reduced when heating is carried out at 850°C than at 900°C. Accordingly, heating at a temperature of 850°C may be preferred in some applications. Moreover, from a comparison of the second through fourth samples, it may be seen that the average amount of wear can be reduced by reducing the oxygen concentration from 10 vol% to 1.7 vol%, so such oxygen concentration reduction also may be preferable in some applications.

[0018] Fig. 4 is a cross sectional diagram of a surface hardened beta titanium member 11 formed according to the methods taught herein. In this condition, beta titanium member 11 comprises a topmost oxidized layer 11a, a hardened oxygen diffusion layer 11b having a thick-

ness of approximately 100 μm below oxidized layer 11a, and an unprocessed layer 11c below hardened layer 11b. Oxidized layer 11a has a thickness of from approximately 0 μm to approximately 5 μm . Such a layer is significantly thinner than the oxidized layers formed in the prior art processes that heat the titanium member in atmospheric air. Thus, removal of oxidized layer 11a created by the teachings herein is very easy.

[0019] In other words, because hardened layer 11b can be formed to a thickness of at least 70 μm (preferably 100 μm) while minimizing the thickness of oxidized layer 11a, a beta titanium member 11 having increased surface hardness can be efficiently obtained. When the same processes as described above are performed in atmospheric air, a hardened layer may be formed to a thickness of 300 μm with an increased HV hardness of 500, but an oxidized layer having a thickness of 100 μm is formed on top of the hardened layer. An oxidized layer on the surface of the product is undesirable because it tarnishes the product's appearance. Since the oxidized layer is hard and brittle, removal of such a thick layer is extremely cumbersome and impairs production efficiency.

[0020] The processes described above have particular benefit when applied to beta titanium members. When the process was applied to pure titanium and alpha-beta titanium alloys, a hardened oxygen diffusion layer did not form. This is thought to be due to the fact that an oxygen diffusion layer cannot be formed via melting of the surface of pure or alpha-beta titanium, whereas such a layer can be formed in beta titanium by surface melting.

[0021] While the above is a description of various embodiments of inventive features, further modifications may be employed without departing from the spirit and scope of the present invention. For example, while argon gas was used solely as the inert gas, other inert gases could be used alone or in combination argon in addition to the oxygen. The size, shape, location or orientation of the various components may be changed as desired. Components that are shown directly connected or contacting each other may have intermediate structures disposed between them. The functions of one element may be performed by two, and vice versa. The structures and functions of one embodiment may be adopted in another embodiment. It is not necessary for all advantages to be present in a particular embodiment at the same time. Every feature which is unique from the prior art, alone or in combination with other features, also should be considered a separate description of further inventions by the applicant, including the structural and/or functional concepts embodied by such feature(s). Thus, the scope of the invention should not be limited by the specific structures disclosed or the apparent initial focus or emphasis on a particular structure or feature.

Claims

1. A method of hardening the surface of a beta titanium member (11) comprising the step of process heating the beta titanium member (11) in a gas mixture consisting essentially of an inert gas and oxygen.
2. The method according to claim 1 wherein the gas mixture has an oxygen concentration in a range of from approximately 0.05 vol% to approximately 20 vol%.
3. The method according to claim 1 or 2 wherein the gas mixture has an oxygen concentration in a range of from approximately 1.0 vol% to approximately 10 vol%.
4. The method according to any of claims 1 to 3 wherein the inert gas comprises argon.
5. The method according to claim 4 wherein the inert gas consists of argon.
6. The method according to any of the preceding claims wherein the process heating step comprises the step of heating the beta titanium member (11) at a temperature from approximately 700°C to approximately 1000°C.
7. The method according to claim 6 wherein the process heating step comprises the step of heating the beta titanium member (11) at a temperature from approximately 700°C to approximately 1000°C for a time period of from approximately 10 minutes to approximately 30 minutes.
8. The method according to claim 7 wherein the time period is from approximately 15 minutes to approximately 25 minutes.
9. The method according to claim 6 wherein the process heating step comprises the step of heating the beta titanium member (11) at a temperature from approximately 850°C to approximately 950°C.
10. The method according to claim 9 wherein the process heating step comprises the step of heating the beta titanium member (11) at a temperature from approximately 850°C to approximately 950°C for a time period of from approximately 10 minutes to approximately 30 minutes.
11. The method according to claim 10 wherein the time period is from approximately 15 minutes to approximately 25 minutes.
12. The method according to any of the preceding

claims further comprising the step of subsequently heating the beta titanium member (11) in a temperature range of from approximately 400°C to approximately 550°C after the process heating step.

13. The method according to claim 12 wherein the subsequent heating step comprises the step of heating the beta titanium member (11) in a temperature range of from approximately 400°C to approximately 550°C for a time period of from approximately 6 hours to approximately 16 hours.

14. The method according to claim 13 wherein the time period is from approximately 10 hours to approximately 14 hours.

15. The method according to claim 12 wherein the subsequent heating step comprises the step of heating the beta titanium member (11) in a temperature range of from approximately 450°C to approximately 500°C.

16. The method according to claim 15 wherein the subsequent heating step comprises the step of heating the beta titanium member (11) in a temperature range of from approximately 450°C to approximately 500°C for a time period of from approximately 6 hours to approximately 16 hours after the process heating step.

17. The method according to claim 16 wherein time period is from approximately 10 hours to approximately 14 hours.

18. A method of hardening the surface of a beta titanium member (11) comprising the steps of:

process heating the beta titanium member (11) in a gas mixture consisting essentially of an inert gas and oxygen at a temperature from approximately 700°C to approximately 1000°C for a time period of from approximately 10 minutes to approximately 30 minutes to form an oxygen diffusion layer (11b); and

subsequently heating the beta titanium member (11) in a temperature range of from approximately 400°C to approximately 550°C for a time period of from approximately 6 hours to approximately 16 hours.

19. The method according to claim 18 wherein the time period for the process heating step is from approximately 15 minutes to approximately 25 minutes, and wherein the time period for the subsequent heating step is from approximately 10 hours to approximately 14 hours.

20. The method according to claim 19 wherein the gas

mixture has an oxygen concentration in a range of from approximately 1.0 vol% to approximately 10 vol%.

21. A method of hardening the surface of a beta titanium member (11) comprising the step of heating the beta titanium member (11) in a gas mixture consisting essentially of an inert gas and oxygen to form a hardened layer (11b) and an oxidized layer (11a) above the hardened layer (11b), wherein the oxidized layer (11a) has a thickness less than or equal to approximately 0.5 µm after the heating has completed.

22. The method according to claim 20 wherein the hardened layer (11b) has a thickness in a range of from approximately 70 µm to approximately 100 µm.

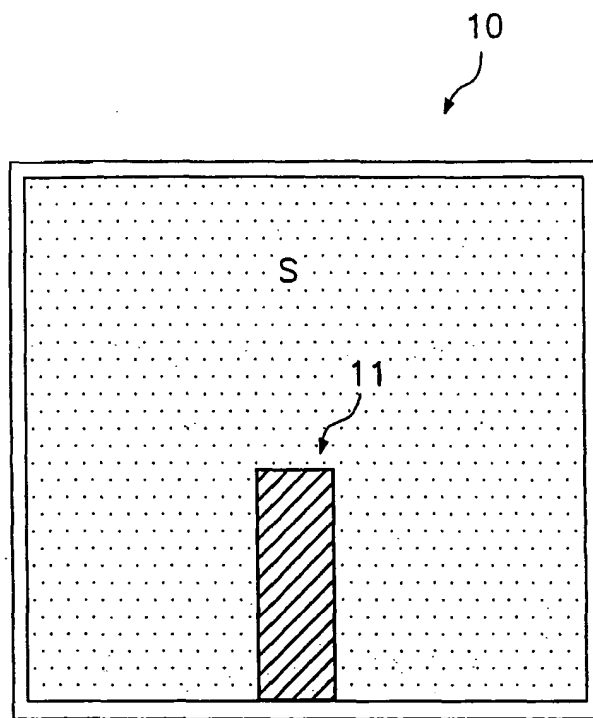


Fig. 1

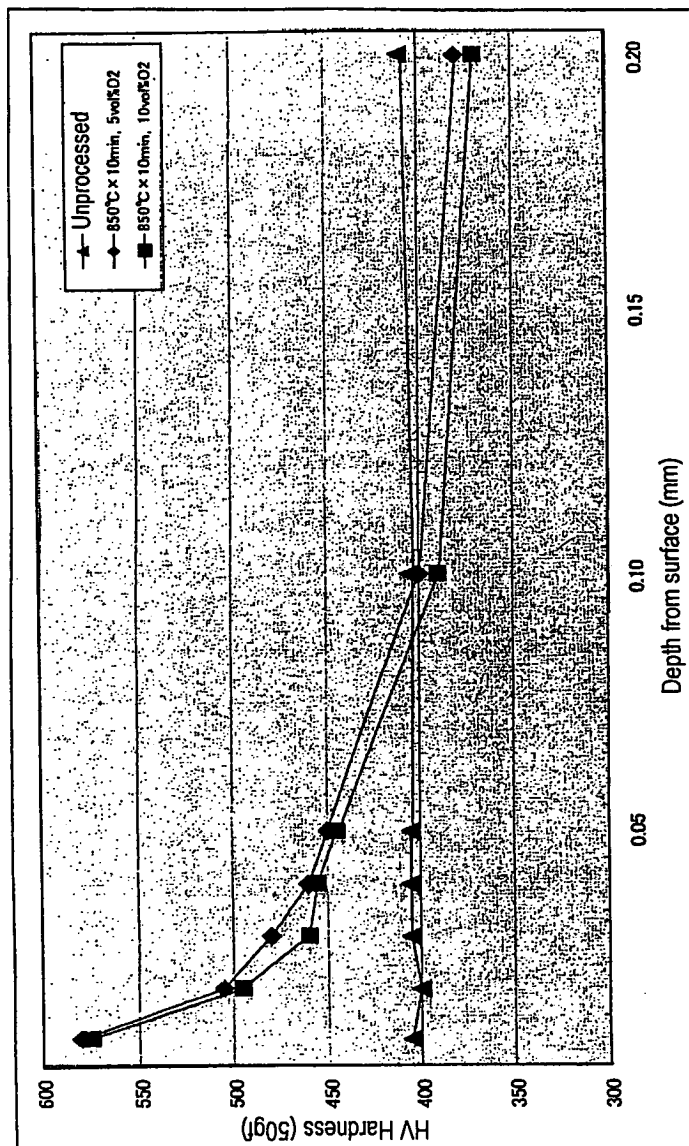


Fig. 2A

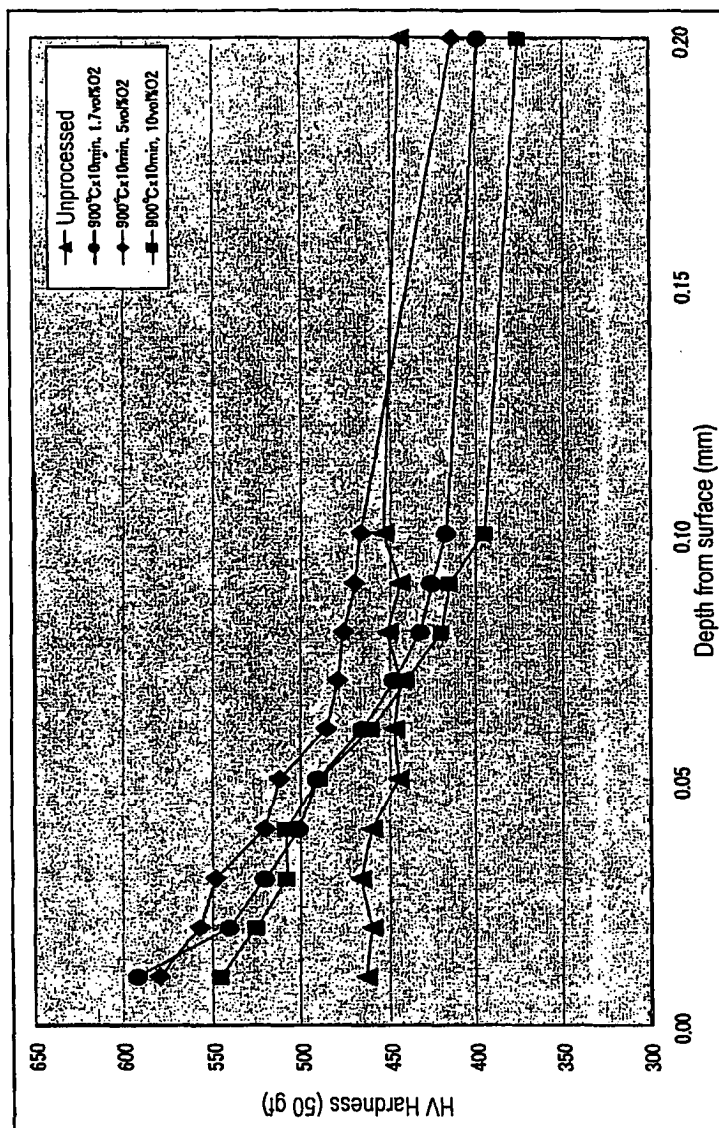


Fig. 2B

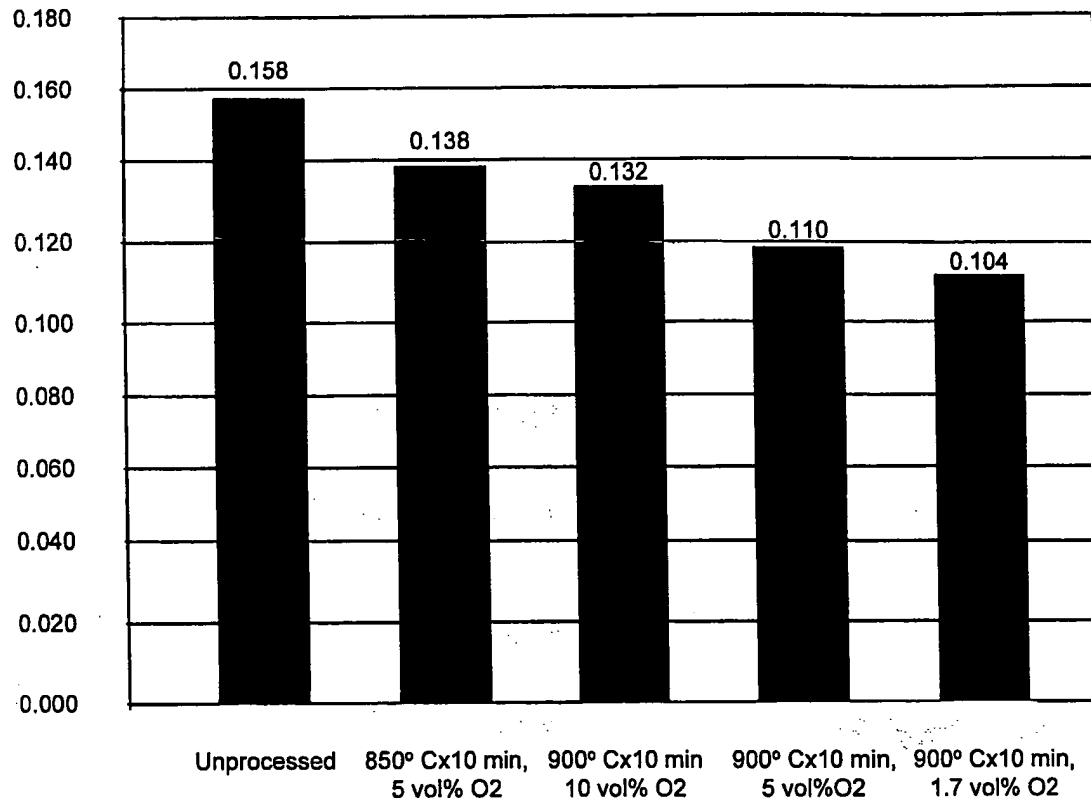


Fig. 3

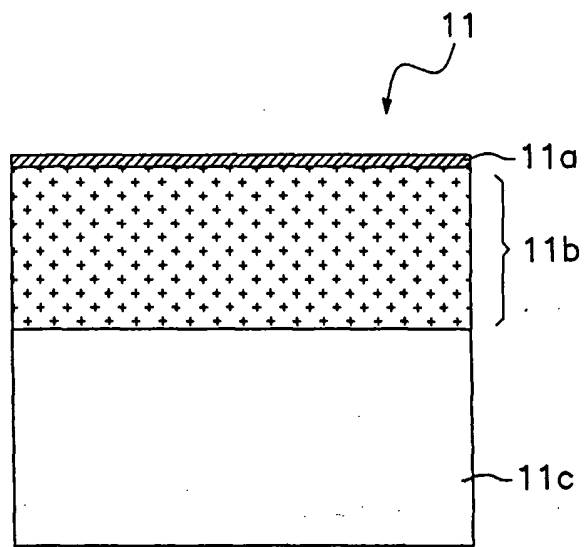


Fig. 4



European Patent
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EUROPEAN SEARCH REPORT

Application Number
EP 05 00 4743

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X	----- EP 1 225 353 A (NSK LTD) 24 July 2002 (2002-07-24) * paragraphs [0034] - [0044], [0101] - [0106], [0123] - [0127], [0239], [0240]; figures 10A-C; table 5 * & WO 02/08623 A (NSK LTD; UEDA, KOJI; UYAMA, HIDEYUKI; SAITO, TSUYOSHI) 31 January 2002 (2002-01-31) -----	1-6,9, 12-17, 21,22	
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The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 12 July 2005	Examiner Hoyer, W
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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**ANNEX TO THE EUROPEAN SEARCH REPORT
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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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