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**Devaux**(10) **Pub. No.: US 2012/0037280 A1**(43) **Pub. Date: Feb. 16, 2012**(54) **METHOD FOR PRODUCING A PART MADE  
FROM A SUPERALLOY BASED ON NICKEL  
AND CORRESPONDING PART**(75) Inventor: **Alexandre Devaux**, Combronde  
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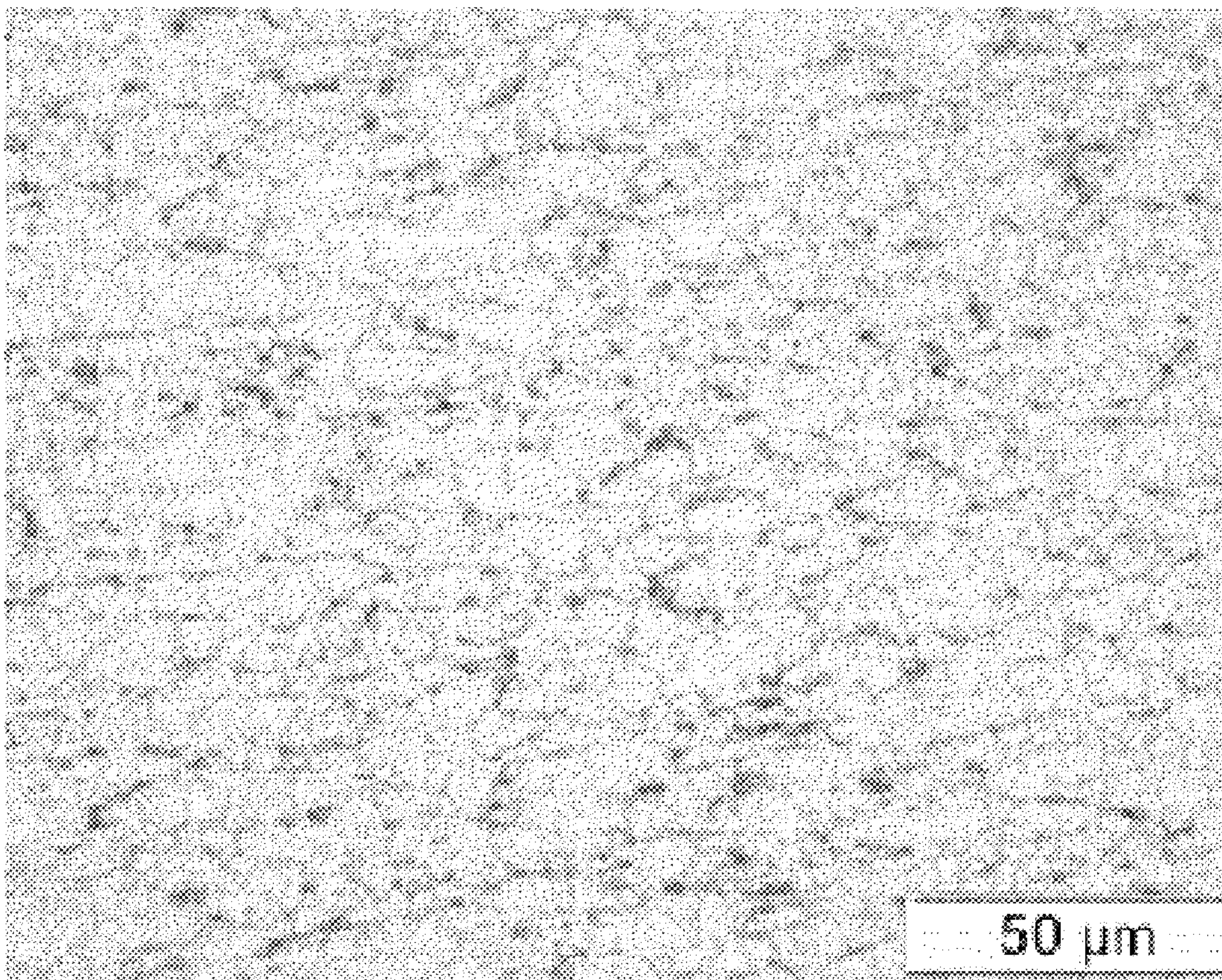
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**ABSTRACT**

A method for manufacturing a blank part in Ni-base superalloy, wherein an alloy is prepared and heat treatments are conducted characterized in that: the said superalloy contains at least a total of 2.5% of Nb and Ta; heat treatment is conducted comprising a plurality of steps: a first step at between 850 and 1000° C. held for at least 20 minutes to precipitate the  $\delta$  phase at the grain boundaries; a second step held at a temperature higher than the temperature of the first step allowing partial dissolution of the  $\delta$  phase obtained at the first step; ageing treatment comprising a third step and optionally one or more additional steps at a temperature below the temperature of the first step and allowing precipitation of the hardening phases  $\gamma'$  and  $\gamma''$ . Part thus obtained.





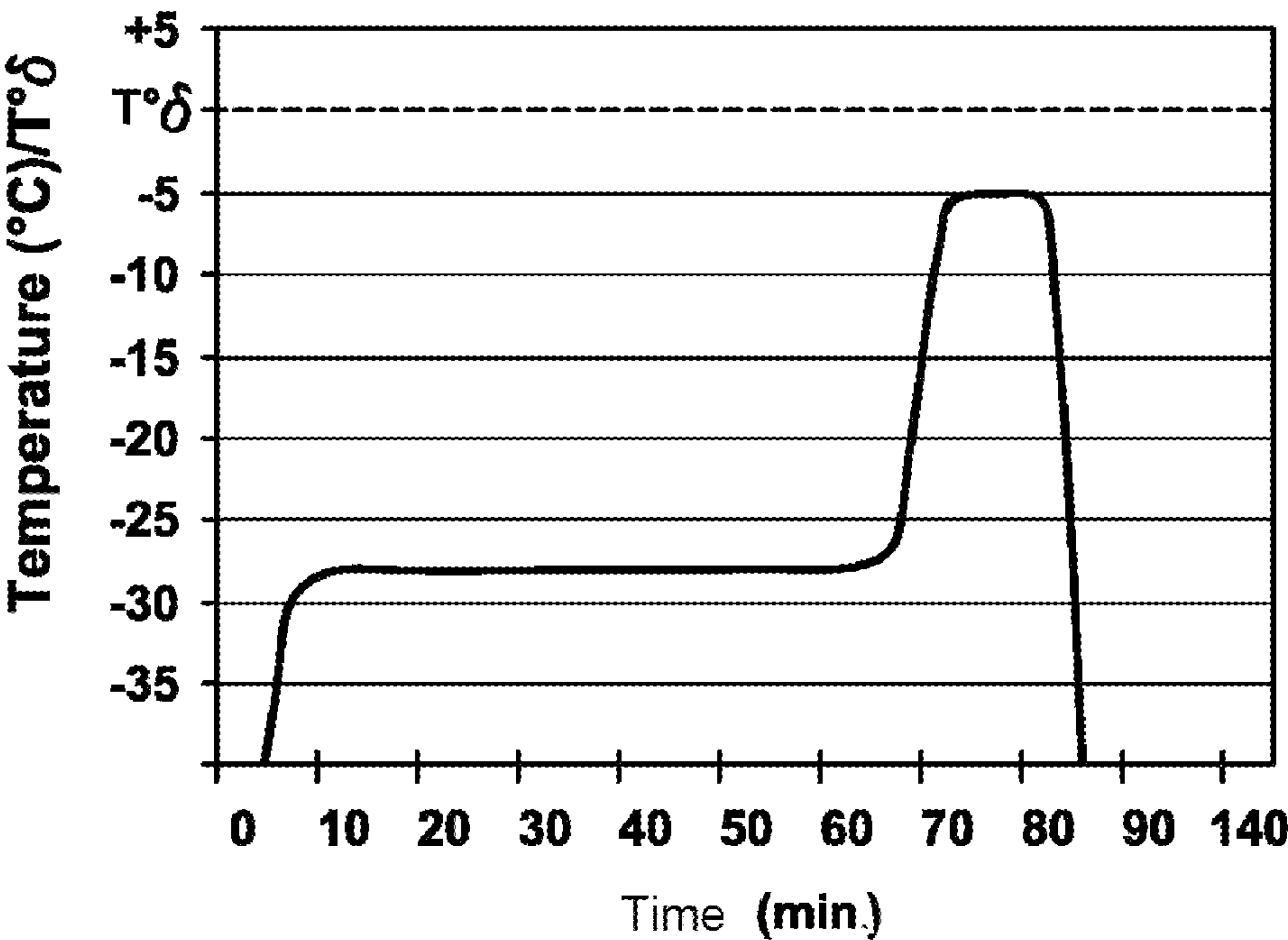


FIG.1

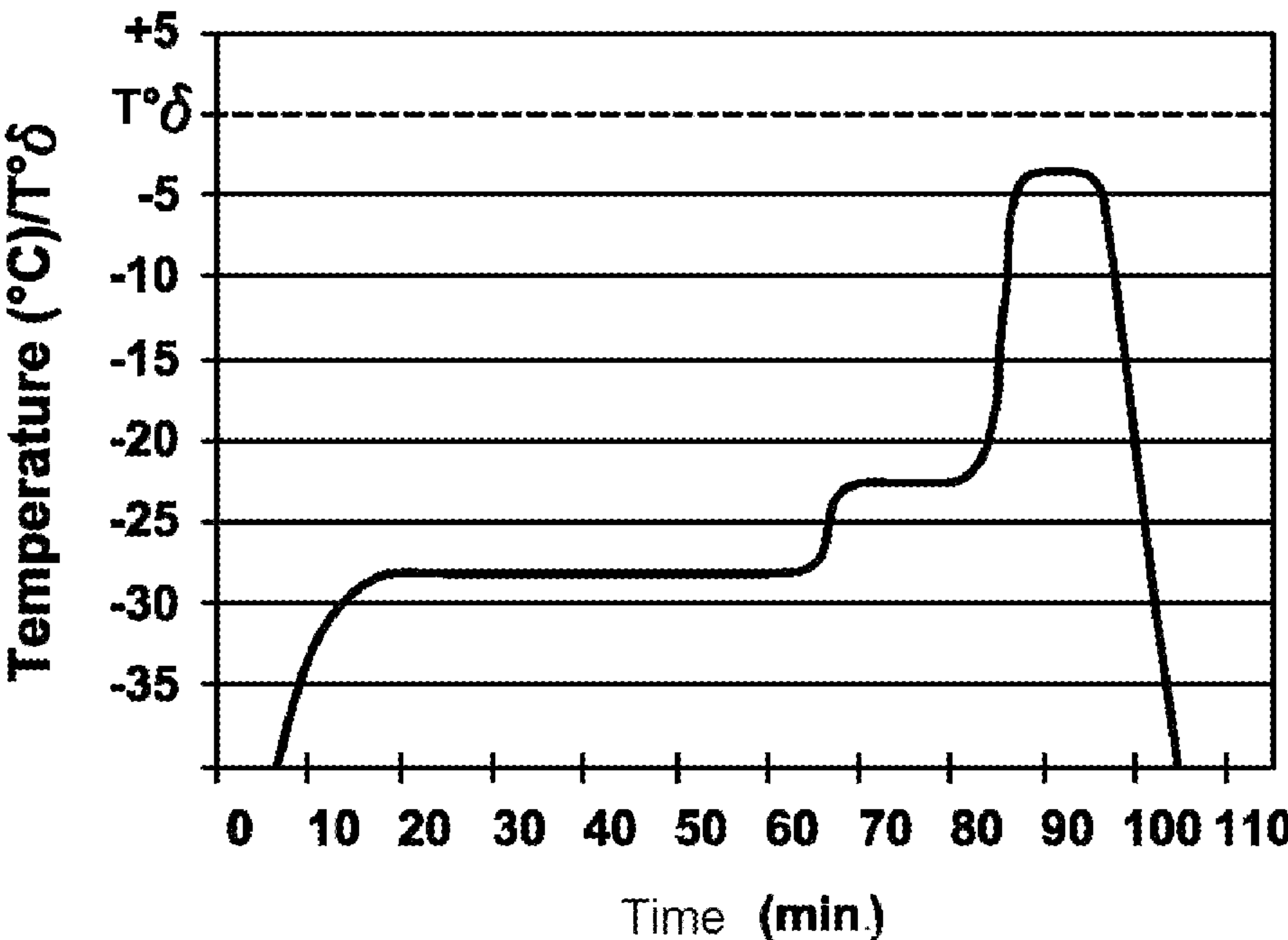


FIG.2

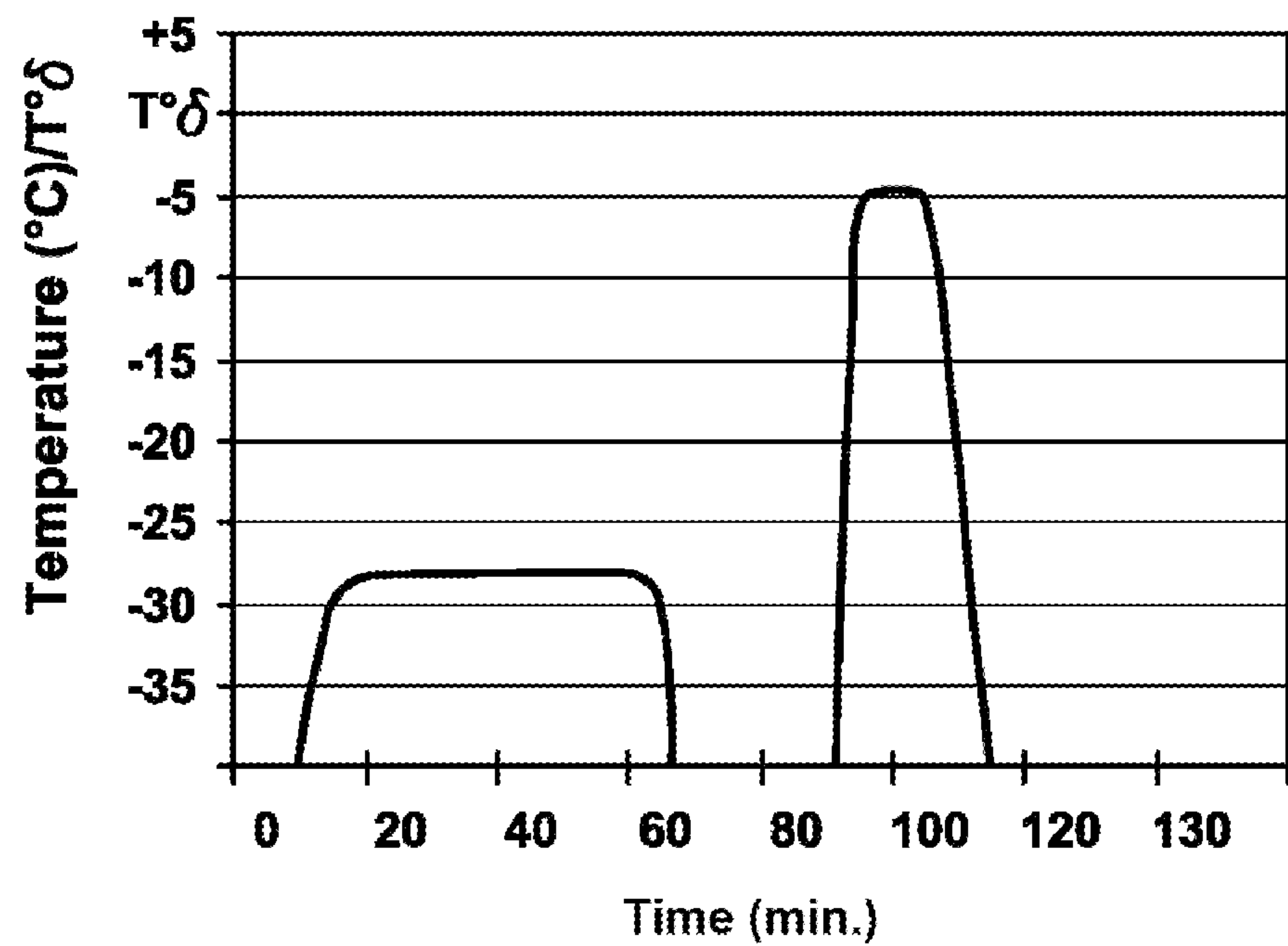


FIG.3

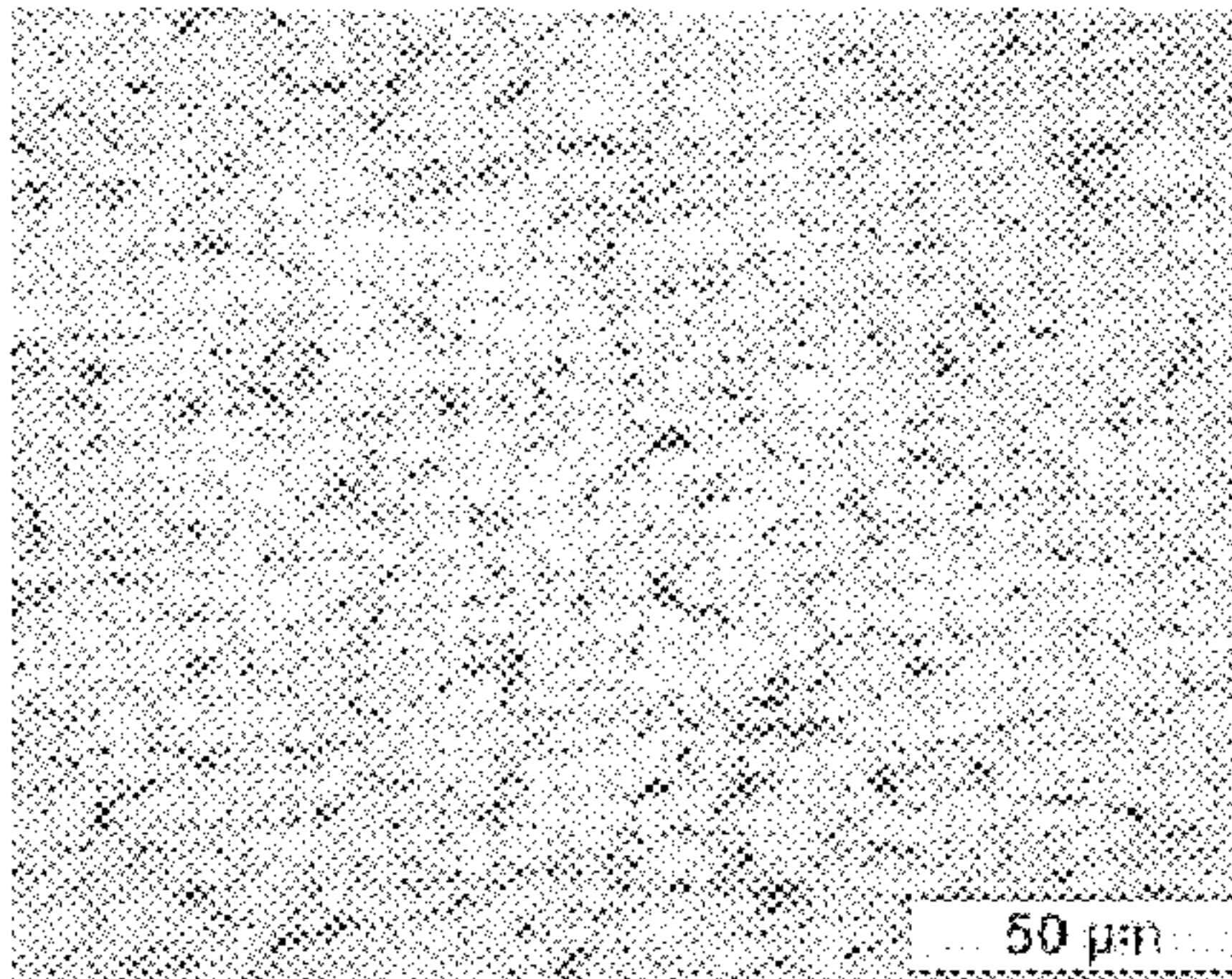


FIG.4

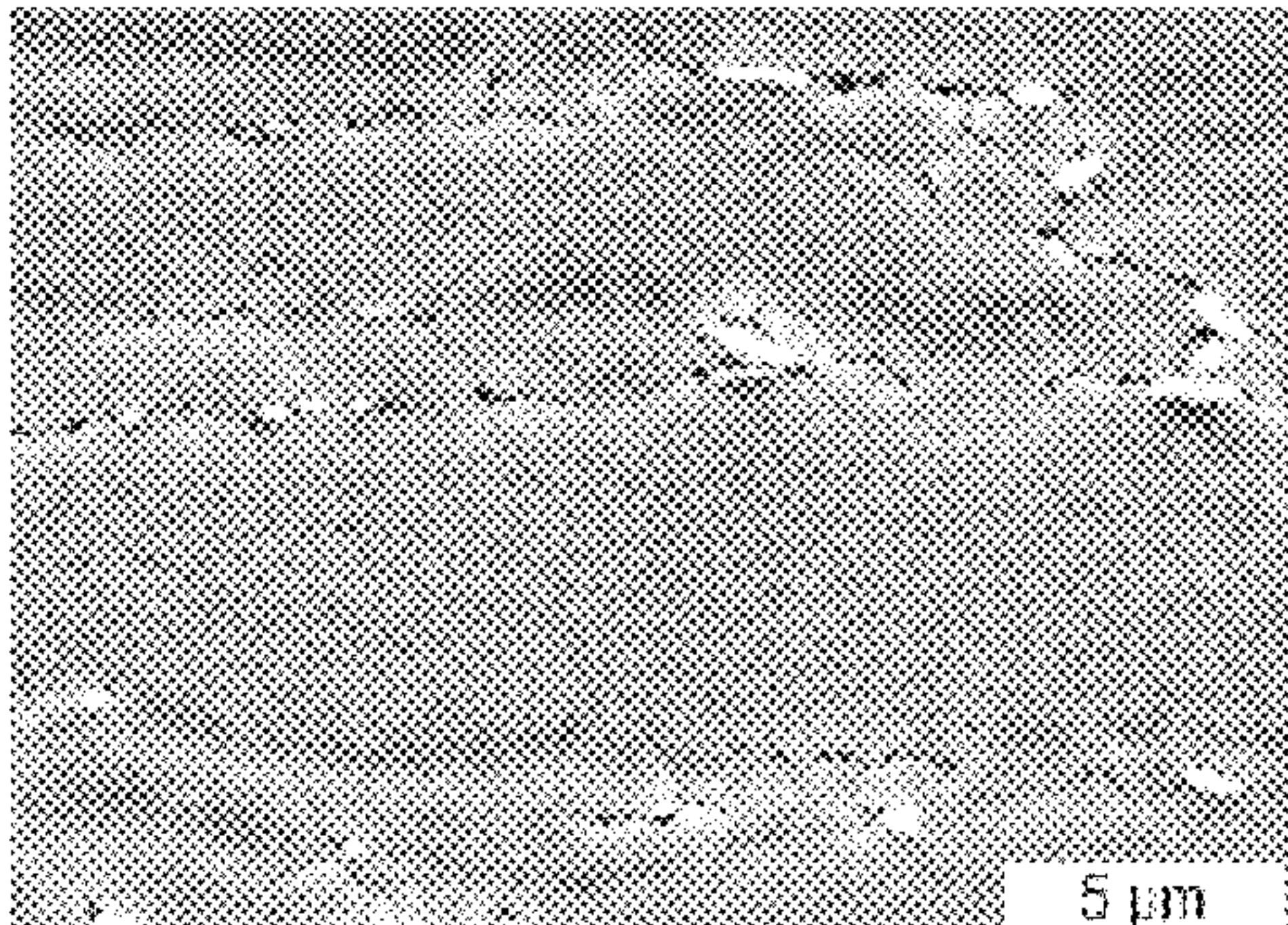


FIG.5



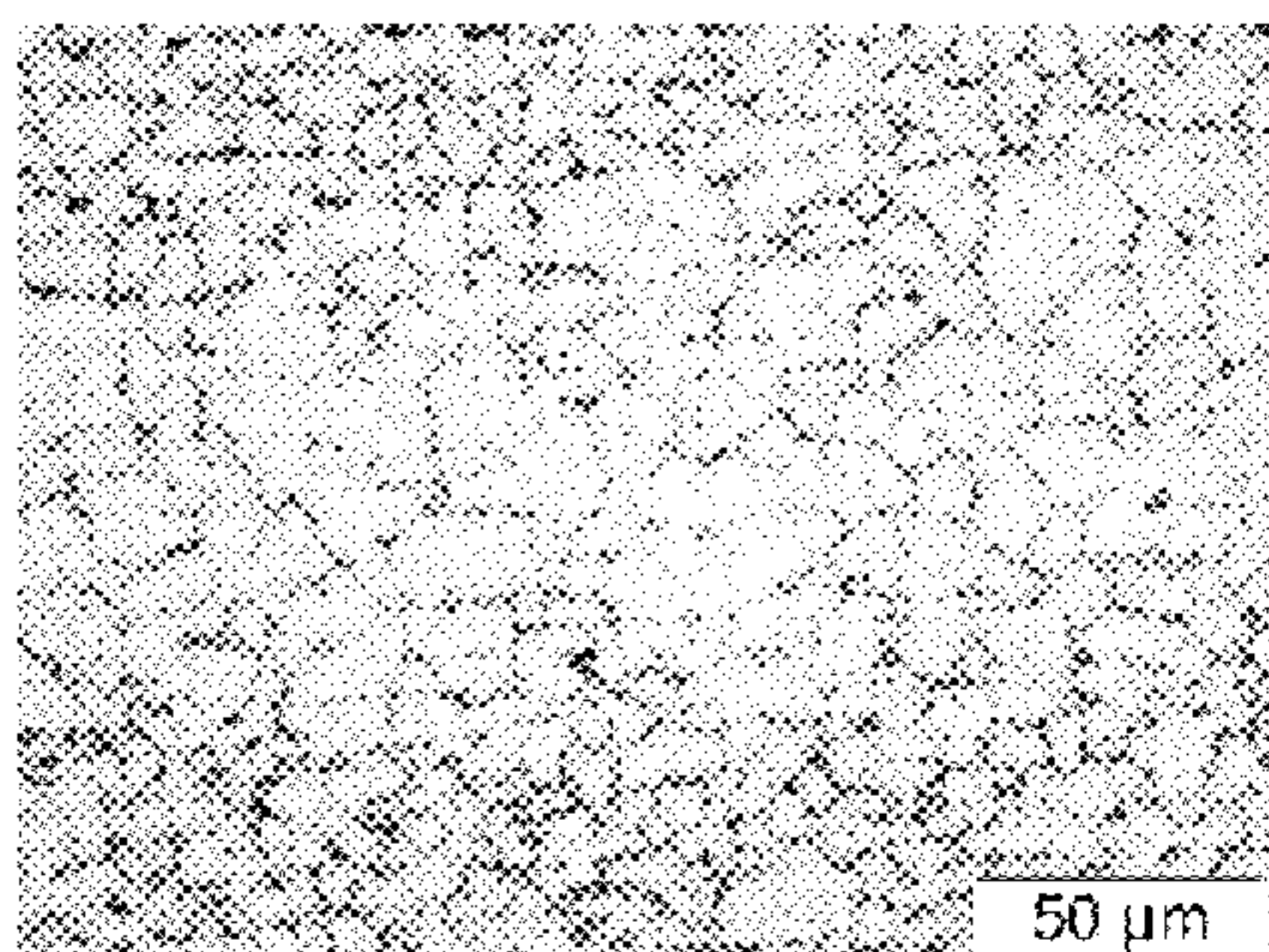


FIG.6

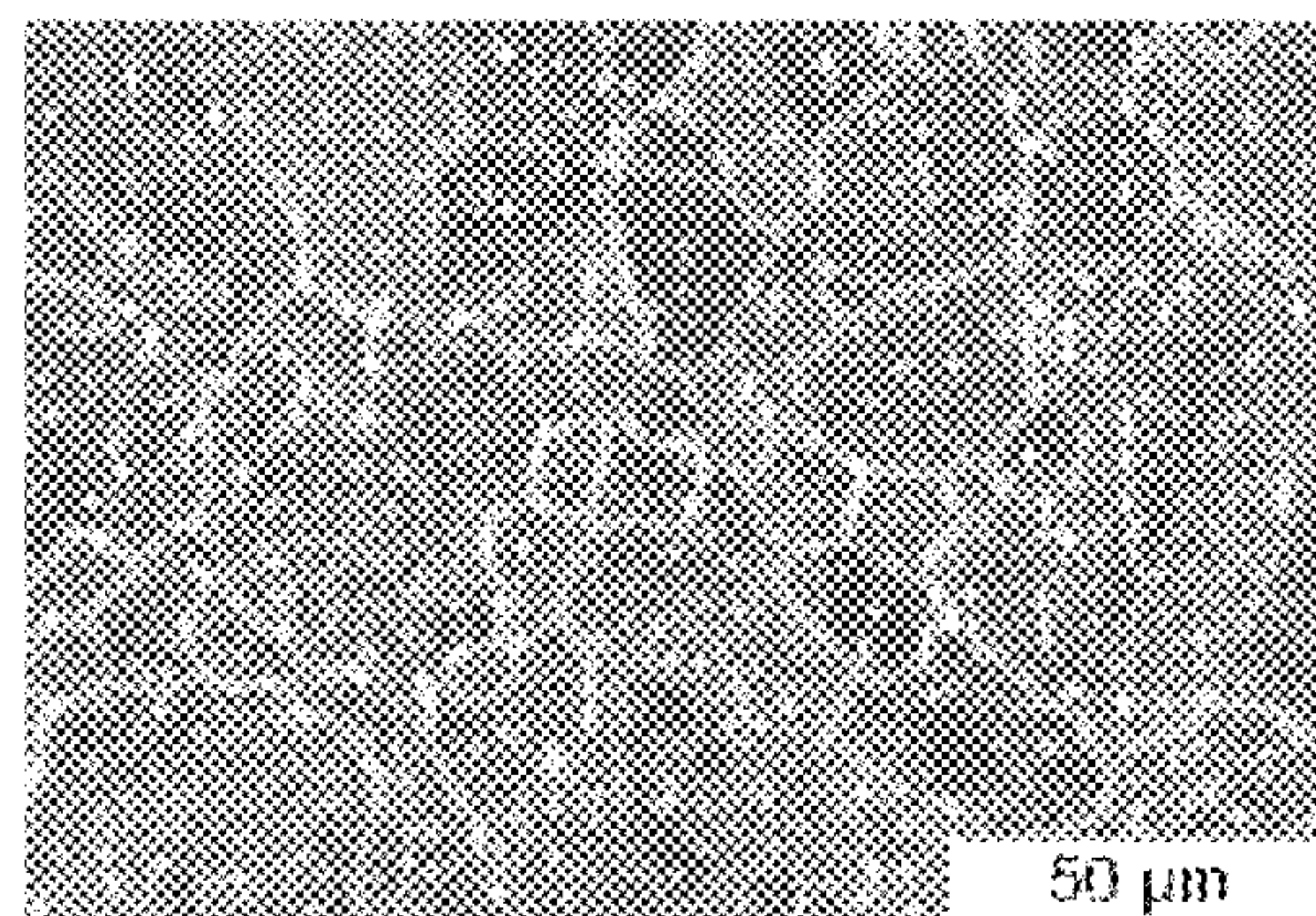


FIG.7

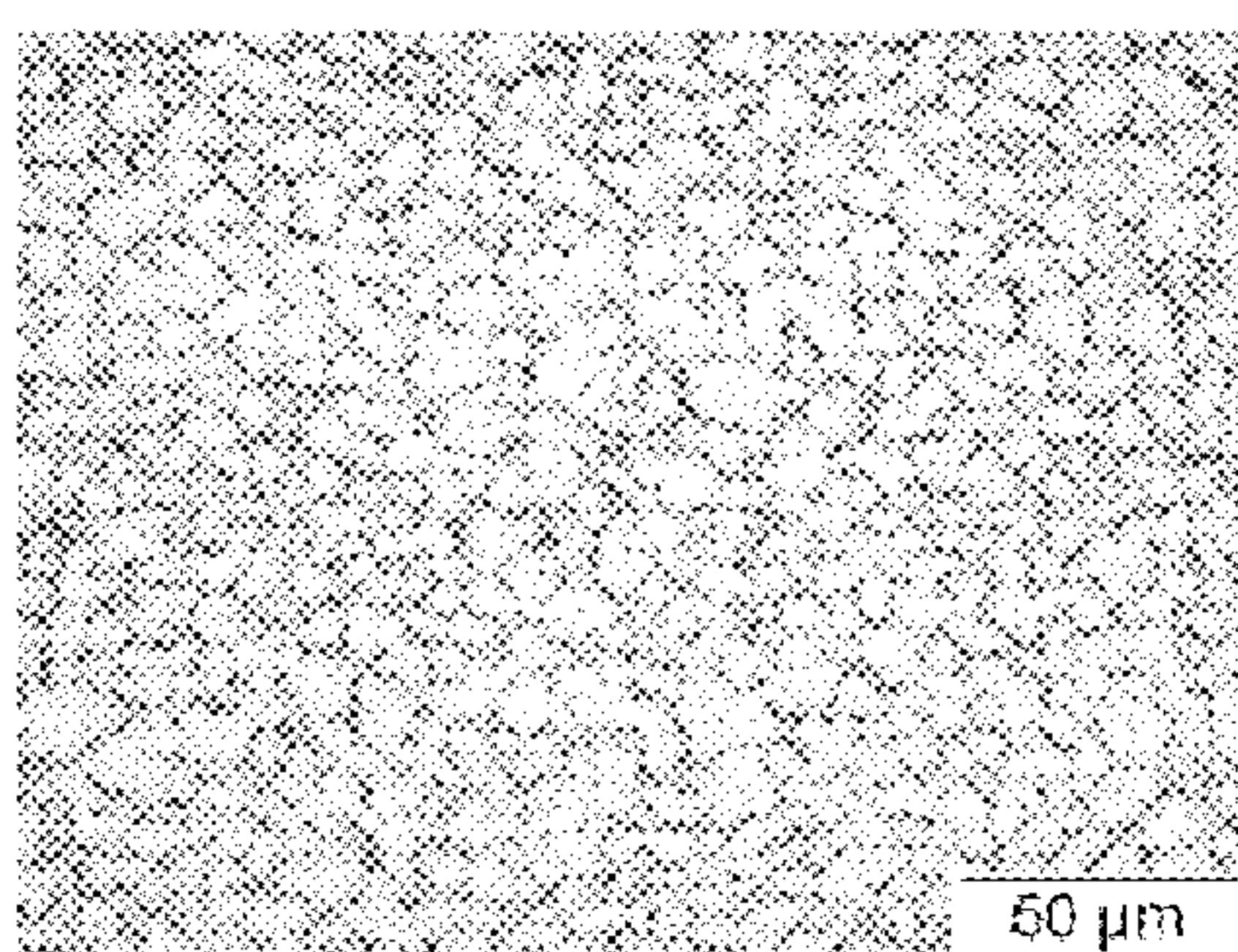


FIG.8

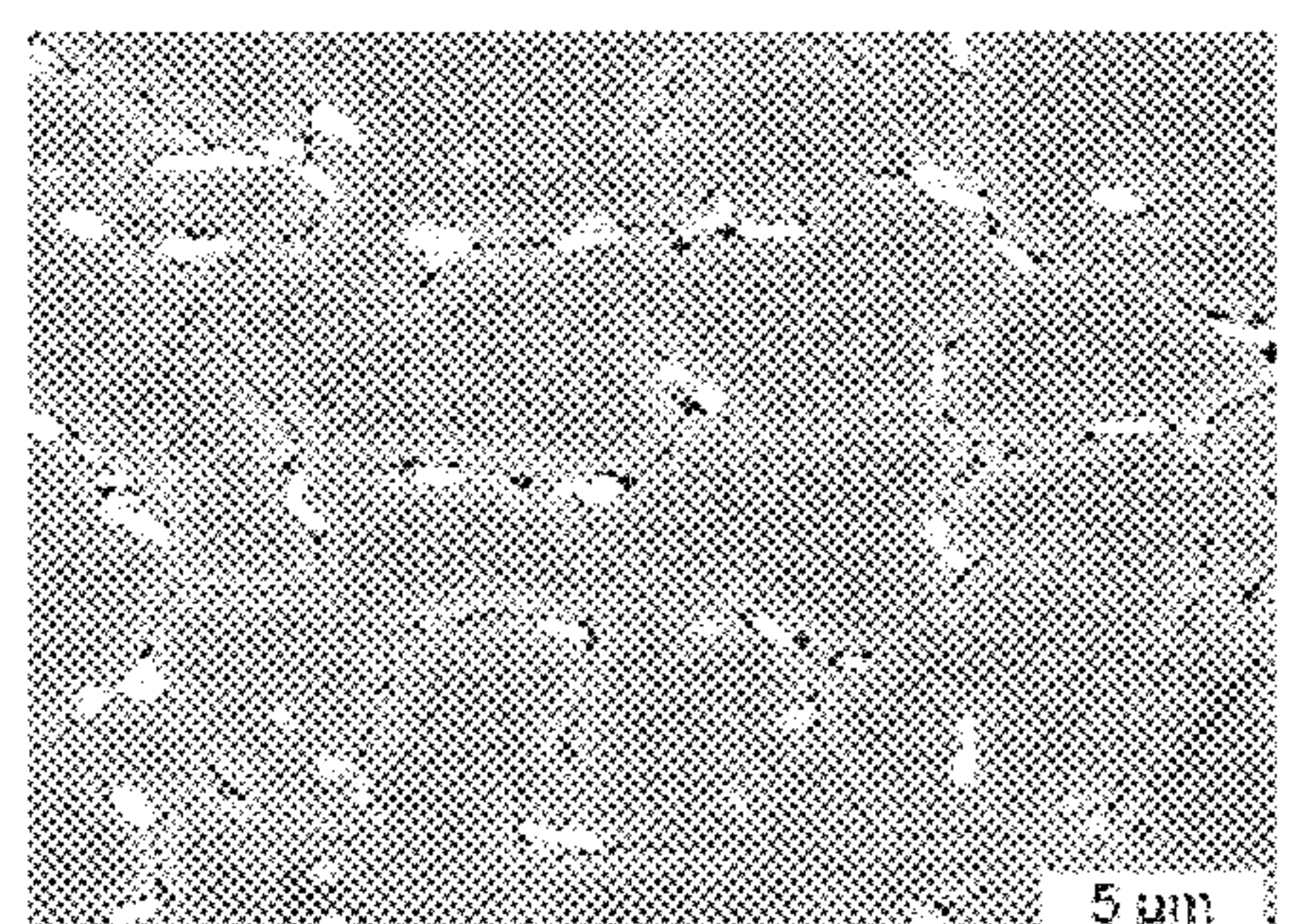


FIG.9



# **METHOD FOR PRODUCING A PART MADE FROM A SUPERALLOY BASED ON NICKEL AND CORRESPONDING PART**

**[0001]** The invention concerns nickel-base superalloys, and more particularly a heat treatment method which can beneficially be applied to some thereof for the improvement in particular of their creep resistance and tensile strength.

**[0002]** By <<nickel-base superalloys>>, is meant alloys in which Ni accounts for at least 50% by weight of the composition (all the percentages given in this text are weight percentages).

**[0003]** More precisely, the invention concerns a heat treatment method applicable to alloys having a content of niobium and tantalum totalling more than 2.5%, and which are therefore able to cause the onset of double precipitation:

**[0004]** intergranular precipitation of the  $\delta$  phase ( $\text{Ni}_3\text{Nb}-\delta$  or  $\text{Ni}_3\text{Ta}-\delta$ ) at between 800 and 1050° C.;

**[0005]** intragranular precipitation of the hardening phases of types  $\gamma'(\text{Ni}_3(\text{Al}-\text{Ti})-\gamma')$  and/or  $\gamma''(\text{Ni}_3\text{Nb}-\gamma''$  or  $\text{Ni}_3\text{Ta}-\gamma'')$  during ageing conducted at between about 600 and 800° C.

**[0006]** This is particularly the case with the alloy NC19FeNb, commercially known as INCONEL 718® (718) and alloys derived therefrom or comparable therewith such as 625, 718Plus and 725.

**[0007]** In the aeronautic and land-based gas turbine industry, in which a nickel-base resistant alloy has numerous applications, experience has shown that the fatigue strength of alloys is among the most critical factors for the sizing of turbine discs and shafts.

**[0008]** The relatively low cost of the 718 alloy, through the absence of cobalt in its composition and the acquired know-how for its production and transformation, give this alloy privileged ranking among the high characteristic alloys used up to a temperature close to 650° C. However, the increased yield and performance of turbo-machines translates as an increase in temperature at the output of the combustion chamber, and therefore requires improved creep resistance of the 718 alloy to increase the possibilities of extending periods of use up to 650° C. The improvement in the creep resistance of the 718 alloy, whilst maintaining a fine grain microstructure (>7 ASTM) so as not to compromise fatigue strength, is therefore of major industrial interest. It is recalled that the ASTM standards governing the estimation of grain size define the grains as being finer the higher the attributed ASTM number.

**[0009]** Two different thermo-mechanical treatment methods are known and currently used to improve the fatigue properties of the 718 alloy.

**[0010]** According to a first option such as described in FR-A-2 089 069, it was chosen to perform thermo-mechanical treatment allowing the  $\text{Ni}_3\text{Nb}-\delta$  phase to be precipitated at the grain boundaries, followed by recrystallization treatment of the alloy at a temperature below the dissolution temperature of the  $\text{Ni}_3\text{Nb}-\delta$  phase, the  $\text{Ni}_3\text{Nb}-\delta$  phase precipitated at the grain boundaries being used during recrystallization to prevent grain growth. With this method it is possible to obtain recrystallized structures with very fine grain size, of ASTM 10 or higher. Their fatigue characteristics are improved but the creep resistance thereof is insufficient. Indeed it is known that the presence of the  $\text{Ni}_3\text{Nb}-\delta$  phase, having an orthorhombic structure, is detrimental since it fixes the niobium and thereby limits the formation of the  $\text{Ni}_3\text{Nb}-\gamma''$  hardening phase, that is metastable and of centred quadratic structure. The

$\text{Ni}_3\text{Nb}-\gamma''$  hardening phase allows the slowing of dislocation movement within the crystallographic lattice and thereby improves creep resistance.

**[0011]** Similarly, it is also known that the presence of the  $\text{Ni}_3\text{Ta}-\delta$  phase is detrimental, since it fixes the tantalum and therefore limits the formation of the  $\text{Ni}_3\text{Ta}-\gamma''$  hardening phase.

**[0012]** Another known solution for improving the properties of 718 consists of conducting ageing directly after thermo-mechanical treatment i.e. without the usual solution heat treatment at between 900 and 980° C. carried out between the thermo-mechanical treatment and the ageing treatment. Although this option allows limited formation of the  $\text{Ni}_3\text{Nb}-\delta$  phase which may precipitate during the solution heat treatment, and the obtaining of fine grain size together with improved tensile and fatigue properties, it does have disadvantages.

**[0013]** It has been found that heterogeneous microstructures are obtained within one same part owing to major local variations in grain size and to the proportion of  $\delta$  phase formed during thermo-mechanical treatments.

**[0014]** As a result, creep resistance is degraded compared with prior practice over a wide temperature and stress range.

**[0015]** Document EP-A-1 398 393 describes treatments of Ni-base superalloys in the form of directionally solidified single crystals or alloys. If the alloy is a single crystal there is evidently no precipitation of  $\delta$  phase at the grain boundaries since there are no grain boundaries. With directional solidification, any precipitation of  $\delta$  phase could only occur heterogeneously and would not prevent grain growth. At the end of the treatment, the grains would be too large in size. In addition, the alloy compositions preferably described in this document would not allow precipitation of the  $\delta$  phase, having regard to the Ti, Ta, Nb and Al contents thereof, since this phase would not be stable on account of the high Al content.

**[0016]** Document U.S. Pat. No. 4,459,160 also describes single crystal Ni-base superalloys in which no precipitation of  $\delta$  phase can be observed at the grain boundaries.

**[0017]** It is the objective of the invention to improve the creep resistance and tensile strength of nickel-base superalloys having a content of niobium and/or tantalum higher than 2.5%, without deteriorating the fatigue properties and whilst avoiding the disadvantages of the aforementioned prior art.

**[0018]** For this purpose, the subject of the invention is a method for manufacturing a Ni-base superalloy blank containing at least 50% Ni as weight percentage, according to which an alloy of said superalloy is produced and the said alloy is subjected to heat treatments, characterized in that:

**[0019]** the said superalloy, in weight percentage, contains Nb and Ta at least to a total amount of 2.5%;

**[0020]** the said alloy is subjected to heat treatment comprising a plurality of hold steps distributed as follows:

**[0021]** a first hold step during which said alloy is held at between 850 and 1000° C. for at least 20 minutes to precipitate the  $\delta$  phase at the grain boundaries;

**[0022]** a second hold step during which said alloy is held at a temperature higher than the temperature of the first hold step and allowing partial dissolution of the  $\delta$  phase obtained at the first step;

**[0023]** aging treatment comprising a third step and optionally one or more additional steps conducted at a temperature lower than that of the first step and allowing precipitation of the hardening phases  $\gamma'$  and/or  $\gamma''$

**[0024]** Preferably, the Al content of the alloy is equal to or less than 3%.

**[0025]** Preferably, the  $(\text{Nb}+\text{Ta}+\text{Ti})/\text{Al}$  ratio of the alloy is 3 or higher.



[0026] Preferably, the grain size obtained at the end of the aging treatment of the alloy ranges from 7 to 13 ASTM, more preferably from 8 to 12 ASTM, further preferably from 9 to 11 ASTM.

[0027] Preferably, the distribution of the  $\delta$  phase is homogeneous at the grain boundaries on completion of the ageing treatment.

[0028] After the second hold step, preferably a quantity of  $\delta$  phase is obtained of between 2 and 4%, and is best between 2.5 and 3.5%.

[0029] The first and second hold steps are preferably conducted without intermediate cooling.

[0030] The changeover from the first to the second hold step can then take place at a rate of 4° C./min or less, preferably between 1 and 3° C./min.

[0031] The first hold step can be conducted at between 900 and 1000° C. for at least 30 min, and the second hold step at between 940 and 1020° C. for 5 to 90 min, the temperature difference between the two temperature holds being at least 20° C.

[0032] The content by weight of the alloy may be as follows:

[0033] between 50 and 55% nickel,

[0034] between 17 and 21% chromium,

[0035] less than 0.08% carbon,

[0036] less than 0.35% manganese,

[0037] less than 1% cobalt,

[0038] less than 0.35% silicon,

[0039] between 2.8 and 3.3% molybdenum,

[0040] at least one of the elements niobium or tantalum, such that the sum of niobium and tantalum totals between 4.75% and 5.5% with Ta being less than 0.2%,

[0041] between 0.65 and 1.15% titanium,

[0042] between 0.20 and 0.80% aluminium,

[0043] less than 0.006% boron,

[0044] less than 0.015% phosphorus,

[0045] the residual percentage being iron and impurities resulting from processing.

[0046] The first hold step can then be conducted at between 920 and 990° C. for at least 30 min, and the second hold step at a temperature of between 960 and 1010° C. for 5 to 45 min.

[0047] The total content of Nb and Ta of the alloy may then be between 5.2 and 5.5%, the first hold step being conducted at between 960 and 990° C. for 45 min to 2 h and the second hold step at between 990 and 1010° C. for 5 to 45 min.

[0048] If the total content of Nb and Ta of the alloy is between 4.8 and 5.2%, the first hold step can be conducted at between 920 and 960° C. for 45 min to 2 h and the second hold step can be conducted at between 960 and 990° C. for 5 to 45 min.

[0049] The content by weight of the alloy may be:

[0050] between 55 and 61% nickel,

[0051] between 19 and 22.5% chromium,

[0052] between 7 and 9.5% molybdenum,

[0053] at least one of the elements niobium or tantalum, such that the sum of niobium and tantalum totals between 2.75 and 4% with Ta being less than 0.2%,

[0054] between 1 and 1.7% titanium,

[0055] less than 0.55% aluminium,

[0056] less than 0.5% cobalt,

[0057] less than 0.03% carbon,

[0058] less than 0.35% manganese,

[0059] less than 0.2% silicon,

[0060] less than 0.006% boron,

[0061] less than 0.015% phosphorus,

[0062] less than 0.01% sulphur,

[0063] the residual percentage being iron and impurities resulting from processing.

[0064] The alloy may have a weight content of:

[0065] between 12 and 20% chromium,

[0066] between 2 and 4% molybdenum,

[0067] at least one of the elements niobium or tantalum, such that the sum of niobium or tantalum is between 5 and 7% with Ta less than 0.2%,

[0068] between 1 and 2% tungsten,

[0069] between 5 and 10% cobalt,

[0070] between 0.4 and 1.4% titanium,

[0071] between 0.6 and 2.6% aluminium,

[0072] between 6 and 14% iron,

[0073] less than 0.1% carbon,

[0074] less than 0.015% boron,

[0075] less than 0.03% phosphorus

[0076] the residual percentage being nickel and impurities resulting from processing.

[0077] Preferably the aforementioned alloys, in weight percentage, have a phosphorus content of more than 0.007%.

[0078] In general, the first and the second hold steps can be conducted at  $\delta$  phase sub-solvus temperatures of the alloy, the first hold step being conducted at a temperature of between 50° C. below the  $\delta$  solvus temperature and 20° C. below the  $\delta$  solvus temperature, and the second hold step being conducted at a temperature of between 20° C. below the  $\delta$  solvus temperature and the  $\delta$  solvus temperature.

[0079] The temperature of the hot-formed blank can be held constant during at least one of the steps.

[0080] The said third step can be conducted at between 700 and 750° C. for 4 to 16 h and a fourth step is then conducted at between 600 and 650° C. for between 4 and 16 h, cooling at 50° C./h to  $\pm 10^\circ$  C./h being conducted between said third and fourth steps.

[0081] Between the first and second steps, it is possible to hold the hot-formed alloy at an intermediate temperature between the temperatures of the first and second steps for a maximum time of 1 h.

[0082] The said blank may have been produced in the form of an ingot and then hot-worked.

[0083] The said blank may have been produced using a powder metallurgy method.

[0084] A further subject of the invention is a part in a nickel-base superalloy, characterized in that it was obtained from a blank produced according to the above-mentioned method.

[0085] This may be a blank of a part for an aeronautic or land-based gas turbine.

[0086] As will have been understood, the invention consists of subjecting a Ni-base alloy containing Nb and/or Ta to a heat treatment for which structural hardening is obtained by precipitation of the gamma' ( $\text{Ni}_3\text{Ti}-\gamma'$ ) and/or gamma'' ( $\text{Ni}_3\text{Nb}-\gamma''$  and/or  $\text{Ni}_3\text{Ta}-\gamma''$ ) hardening phases, these phases respectively comprising Titanium and Niobium and/or Tantalum. The heat treatment comprises at least three hold steps which chronologically are the following:

[0087] a first hold step conducted at 850-1000° C. which is intended to precipitate the delta phase  $\text{Ni}_3\text{Nb}-\delta$  and/or  $\text{Ni}_3\text{Ta}-\delta$  at the grain boundaries, with substantially homogeneous distribution of this phase in the grain boundaries, and to homogenize the microstructure of the material; with regard to partly recrystallized microstructures it also allows completion of recrystallization and causes the  $\delta$  phase to precipitate at the boundaries of the new recrystallized grains;

[0088] a second hold step conducted at a temperature higher than that of the first step and intended for part



dissolution of the said delta phase  $\text{Ni}_3\text{Nb}-\delta$  and/or  $\text{Ni}_3\text{Ta}-\delta$ , whilst maintaining the substantially homogeneous distribution obtained after the first step, and avoiding grain enlargement; the second step is completed by oil quench or air cooling;

**[0089]** the third step and any optional following steps are thermal ageing steps conducted at a temperature below the temperature of the first step and allowing precipitation of the gamma' ( $\text{Ni}_3(\text{Al}-\text{Ti})-\gamma'$ ) and/or gamma'' ( $\text{Ni}_3\text{Nb}-\gamma''$  or  $\text{Ni}_3\text{Ta}-\gamma''$ ) hardening phases.

**[0090]** One or more intermediate cooling operations are possible between each step but are not compulsory.

**[0091]** The method of the invention allows parts to be produced which, compared with those of the prior art having the same composition, offer a better compromise between yield strength under heavy loading, high fatigue strength and long creep resistance lifetime.

**[0092]** The invention will be better understood on reading the following description given with reference to the following appended figures:

**[0093]** FIGS. 1 to 3 which schematize three examples of the two first heat treatment steps according to the invention, FIG. 2 also showing an intermediate step between the first and second steps; the temperatures along the Y-axis are referenced in relation to  $\delta$  phase solvus temperature.

**[0094]** FIGS. 4 to 9 which give micrographs of alloys subjected to reference heat treatments (FIGS. 4 to 7) and according to the invention (FIGS. 8, 9).

**[0095]** The method for manufacturing a part in Ni superalloy according to the invention may be initiated by preparing and casting an ingot of said superalloy using conventional methods such as a double melt method (VIM Vacuum Induction Melting—VAR Vacuum Arc Remelting) or triple melt (VIM—ESR (Electroslag remelting)—VAR). However, the method of the invention can also be applied to a blank produced by power metallurgy. In the remainder of the text the examples of applications described are examples in which the starting product is obtained by the conventional route called <<ingot metallurgy>>, but the transposition thereof to powder metallurgy will be obvious for persons skilled in the art. The treatments following after hot-working that are characteristic of the invention will be the same in both cases.

**[0096]** The initial microstructure of a product (on the understanding that the term <<product>> designates a semi-product or blank of a part) before the treatment typical of the invention, may vary in relation to the deformation thermo-mechanical treatments conducted upstream, for example forging, punching or hot rolling:

**[0097]** metallurgical state 1 (or <<state 1>>): the delta phase  $\text{Ni}_3\text{Nb}-\delta$  and/or  $\text{Ni}_3\text{Ta}-\delta$  can be present at the grain boundaries but not uniformly distributed between the grains subsequent to deformation conducted at a temperature lower than  $\delta$  phase solvus temperature;

**[0098]** metallurgical state 2 (or <<state 2>>): the delta phase  $\text{Ni}_3\text{Nb}-\delta$  and/or  $\text{Ni}_3\text{Ta}-\delta$  may be absent or practically absent (<1%) from the microstructure subsequent to deformation conducted for example at a temperature higher than the  $\delta$  phase solvus.

**[0099]** In the first case, i.e. starting from metallurgical state 1, the first treatment step according to the invention allows the distribution of the  $\delta$  phase to be homogenized within the microstructure, and permits the reducing of local variations in the  $\delta$  phase fraction present after the thermo-mechanical treatments due to temperature variations of greater or lesser extent after deformation. Persons skilled in the art, if necessary, are easily able to adjust the parameters for conducting

the first step through routine testing, in order to optimize this homogenization of  $\delta$  phase distribution.

**[0100]** In the second case i.e. starting from metallurgical state 2, the first treatment step according to the invention allows (substantially) homogeneous precipitation of  $\delta$  phase at the grain boundaries which were devoid of this phase after the thermo-mechanical treatment. Persons skilled in the art may also, through routine testing, adjust the parameters if necessary for conducting the first step so as to optimize this homogenization of  $\delta$  phase distribution.

**[0101]** Whether in the first or second case, the first step also allows completion of recrystallization in the regions where recrystallization may not have been complete during thermo-mechanical treatment, and it thereby homogenizes the global structure of the alloy.

**[0102]** At the second step of the treatment according to the invention, conducted at a temperature close to the  $\delta$  phase solvus, the delta phase  $\text{Ni}_3\text{Nb}-\delta$  and/or  $\text{Ni}_3\text{Ta}-\delta$  is partly dissolved.

**[0103]** At the second step, the dissolution of the  $\delta$  phase takes place in substantially uniform manner. The so-called residual  $\delta$  phase i.e. the non-dissolved  $\delta$  phase maintains the same distribution as obtained after the first step. On this account, the residual  $\delta$  phase remains substantially uniformly distributed around the grains, allowing the slowed growth of all the grains and the limiting and even the avoiding of the onset of large grains at the second step, which is conducted at a temperature higher than that of the first step. The homogeneous distribution of the  $\delta$  phase at the grain boundaries promotes the homogeneity of grain size in the microstructure of the alloy at the end of treatment.

**[0104]** The second step therefore allows a reduction in the quantity of  $\delta$  phase obtained after the first step, down to a residual quantity that is optimally lower than 4%, even below 3.5% whilst avoiding grain enlargement.

**[0105]** The greater dissolution of the  $\delta$  phase in a fine-grain, homogeneous microstructure allows more niobium to be released for precipitation of the gamma' and/or gamma'' hardening phases during a third step and even other subsequent steps forming an ageing treatment of the alloy.

**[0106]** In unexpected manner, the inventors have found that the absence of the first treatment step does not allow these effects to be obtained, irrespective of the initial microstructure after thermo-mechanical treatment.

**[0107]** Evidently, for an initial microstructure devoid of  $\delta$  phase (state 2) the absence of the first step will not allow homogenization of the global structure of the material and precipitation of the  $\delta$  phase at the grain boundaries, limiting subsequent growth of the grains during the second step.

**[0108]** In the absence of the first step, when the initial microstructure results from sub-solvus deformation which led to  $\delta$  phase precipitation (state 1), the distribution of the  $\delta$  phase is heterogeneous (see FIGS. 4 and 5). Therefore some grains may contain a large quantity of  $\delta$  phase at the grain boundaries, or little or no  $\delta$  phase at the grain boundaries, or even a heterogeneous distribution of  $\delta$  phase at the grain boundaries.

**[0109]** By conducting a heat treatment directly at the temperature of the second step, without a temperature hold at the temperature of the first step, the grains which are not surrounded with  $\delta$  phase or which have little  $\delta$  phase at the grain boundaries, or a non-uniformly distributed  $\delta$  phase will enlarge uncontrollably up to a grain size possibly exceeding about ASTM 5-6. The presence, even much localised presence of ASTM 5-6 grains (see FIGS. 6 and 7), reduces the fatigue lifetimes by a factor of 10 compared with a homogeneous microstructure having ASTM size 10 grains. The com-



bination of the first and second steps according to the invention therefore (see FIGS. 8 and 9) allow the partial dissolution of the  $\delta$  phase and in homogeneous manner, whilst avoiding the presence of these large ASTM 5-6 grains which is redhibitory for guaranteeing high fatigue properties.

[0110] With an initial microstructure comprising  $\delta$  phase (state 1), the absence of the first step does not therefore allow the desired microstructure to be obtained i.e. having a residual, homogeneous  $\delta$  phase content preferably less than 4% with a homogeneous and acceptable grain size.

[0111] The preferred grain size for the products derived from the method of the invention follows from the desire to achieve a good compromise between conflicting properties with regard to their grain size requirements. Fatigue strength and tensile strength effectively benefit from a small grain size, whereas creep resistance and crack resistance benefit from a coarse grain size. In this perspective, the preferred grain sizes are ASTM 7 to 13, preferably ASTM 8 to 12, and best ASTM 9 to 11.

[0112] The absence of the second step after conducting the first step corresponds to treatments of usual type performed on superalloy products to which the invention applies, and for which it was seen above that they are not satisfactory.

[0113] In addition, for an initial microstructure devoid of  $\delta$  phase (state 2), and if neither of the two first steps required by the invention are conducted, and therefore if thermal ageing treatment is directly applied to the alloy (so-called <<Direct Aged>> treatment) after its hot-working at  $\delta$  phase solvus temperature (state 2), in the final structure a total absence of  $\delta$  phase is obtained which is undesirable.

[0114] Unexpectedly, the inventors were effectively able to evidence that a presence of  $\delta$  phase of between 2 and 4% and optimally between 2.5 and 3.5% allows the properties of the material to improved without weakening thereof.

[0115] On the other hand, microstructures which are devoid of  $\delta$  phase are in general more subject to intergranular weakening which considerably reduces high temperature ductility and strongly increases the alloy's sensitivity to notch effect (for example premature creep rupture at a notched point). Therefore, when the  $\delta$  phase is absent after thermo-mechanical treatment, the first step is also necessary to create a minimum amount of  $\delta$  phase distributed homogeneously at the grain boundaries and to homogenize the global structure of the material.

[0116] The hold period of the alloy at the temperature of the first step is equal to or more than 20 minutes. The temperature of the first step is between 850 and 1000° C. to precipitate the  $\delta$  phase. The temperature and the holding time are adjusted in relation to the heterogeneity of the microstructure after deformation, and with a view to maintaining an amount of  $\delta$  phase after the second step that is higher than the minimum required for hot ductility.

[0117] The second step conducted at a temperature higher than the first step is therefore necessary to allow lowering of the quantity of  $\delta$  phase by dissolution down to the desired level, preferably to a content of between 2 and 4% and optimally between 2.5 and 3.5%, to release the Nb and/or Ta needed for precipitation of the  $\gamma'$  phase and/or  $\gamma''$  phase whilst maintaining a sufficient quantity of Nb and/or Ta in  $\delta$  phase form distributed homogeneously around the grains for the hot ductility of the material.

[0118] The temperature and the duration of the second step are adjusted in relation to the fraction of  $\delta$  phase obtained after the first step in order to obtain the desired residual fraction of  $\delta$  phase, whilst avoiding grain enlargement. The duration of the second step is also related to the temperature

determined for this step. In general, the duration of the second step is shorter the higher the temperature thereof.

[0119] According to one preferred variant of the invention, the two first treatment steps are successive steps (FIGS. 1 and 2).

[0120] By <<successive treatment steps>>, is meant that the changeover from the first treatment step to the second treatment step takes place by progressively increasing the temperature to move from the first step onto the second without passing through an intermediate temperature lower than that of the first step.

[0121] The succession of the two first steps without descending down to a temperature lower than that of the first step, for example down to ambient temperature, allows the avoiding of large temperature gradients inside the treated sample, and the avoiding of heterogeneous dissolution of the  $\delta$  phase which could cause grain enlargement in some regions. It is therefore preferable to adopt a sufficiently low rate of temperature rise (<4° C./min) between the steps so that the temperature remains homogeneous within the treated sample during the second step. It was verified at the second step that the temperature was homogeneous after 5 minutes within a cylindrical sample of 1000 cm<sup>3</sup> after a rate of temperature rise of 2° C./min from the first step. Therefore, any changeover between the two steps at a temperature lower than the first step risks increasing the time needed for homogenization of the temperature within the sample during the second stage, and risks promoting heterogeneous dissolution of the  $\delta$  phase. Nevertheless, said changeover to a temperature lower than that of the first step is not excluded by the invention (FIG. 3) if, in particular in relation to the size of the treated part, the parameters of the second step are adjusted, optionally by adding an intermediate step so as to avoid the possible disadvantages that have just been mentioned.

[0122] Preferably, the first treatment step is conducted at a temperature of between about 900 and 1000° C. for a time of at least 30 minutes, and the second treatment step is conducted at a temperature higher than that of the first step at between 940° and 1020° C. for a time of between 5 and 90 minutes. The difference in temperature between the two steps must therefore be at least 20° C. The temperature ranges and time durations thus obtained allow a homogeneous microstructure to be obtained with an adequate grain size i.e. between ASTM 7 and 13, preferably between ASTM 8 and 12, best between ASTM 9 and 11, and a residual  $\delta$  phase fraction of between 2% and 4%.

[0123] As will have been understood the invention is firstly based on a synergy effect between the two first steps, and the optimised balancing between these two first two steps allows the objectives set by the invention to be best met.

[0124] The  $\delta$  phase solvus temperature is directly dependent upon the niobium+tantalum content of the alloy. The quantity of niobium and/or tantalum present in the composition of the alloy therefore has a direct influence on the temperature and duration of each step.

[0125] If an alloy of type 718 is used (whose standardized composition is detailed below), it is indicated to conduct the first temperature hold at between 920 and 990° C. for at least 30 min, and the second temperature hold at between 960 and 1010° C. for 5 to 45 min. The optimal durations of the treatments are also dependent upon the massiveness of the part to be treated, and can be determined using modelling or experiments usually used by those skilled in the art.

[0126] For a total Nb and Ta content of the 718 alloy (with less than 0.2% Ta) of between 5.2 and 5.5%, the first step is preferably conducted at a temperature of between about 960° C. and 990° C. for a time of between about 45 minutes and 2



hours, and the second step is preferably conducted at a temperature of between about 990° C. and 1010° C. for a time of between about 5 and 45 minutes.

[0127] For a Nb+Ta content of the 718 alloy (with less than 0.2% Ta) of between about 4.8 and 5.2%, the first step is preferably conducted at a temperature of between about 920° C. and 960° C. for a time of between about 45 minutes and 2 hours, and the second step is preferably conducted at a temperature of between 960° C. and 990° C. for a time of between about 5 and 45 minutes. The duration of treatment is also dependent upon the massiveness of the part to be treated.

[0128] The temperatures at the treatment steps are generally held substantially constant throughout the duration of the temperature hold.

[0129] The rate of temperature rise between the first and second step is preferably lower than 4° C./min, to avoid temperature gradients that are too high, especially if the parts being treated are large parts.

[0130] The rate of temperature rise from the first to the second step is preferably between 1° C./min and 3° C./min.

[0131] The invention therefore applies to nickel-base superalloys containing at least 50% Ni, in which the sum of Nb+Ta exceeds 2.5% by weight.

[0132] In one particular case, the alloy is nickel-base alloy of 718 type also called NC19FeNb (AFNOR standard), with a weight content of,

[0133] between 50 and 55% nickel,

[0134] between 17 and 21% chromium,

[0135] less than 0.08% carbon,

[0136] less than 0.35% manganese,

[0137] less than 0.35% silicon,

[0138] less than 1% cobalt,

[0139] between 2.8 and 3.3% molybdenum,

[0140] at least one of the elements niobium or tantalum, such that the sum of niobium and tantalum is between 4.75 and 5.5% with Ta less than 0.2%,

[0141] between 0.65 and 1.15% titanium,

[0142] between 0.20 and 0.80% aluminium,

[0143] less than 0.006% boron,

[0144] less than 0.015% phosphorus,

[0145] the residual percentage being iron and impurities resulting from processing.

[0146] The elements for which no minimum content is given may only be present in trace form, in other words at a content which may be zero, at all events sufficiently low to have no metallurgic effect (this is true for the compositions which will be mentioned).

[0147] Advantageously, an addition of phosphorus allows grain boundary strength to be reinforced, in particular against stresses such as creep and notched creep. The application of the invention to said alloy with phosphorus content higher than 0.007% and lower than 0.015% is of particular interest since the creep gain obtained is distinctly greater. It therefore becomes easily possible to improve creep lifetimes by a factor of 4 whilst maintaining the same grain size. This presence of phosphorus, for the same reasons, may also be recommended for the other examples of alloy give below.

[0148] In another particular case, the alloy is a nickel-base superalloy of 725 type, having a weight content of:

[0149] between 55 and 61% nickel,

[0150] between 19 and 22.5% chromium,

[0151] between 7 and 9.5% molybdenum,

[0152] at least one of the elements niobium or tantalum, such that the sum of niobium and tantalum is between 2.75 and 4% with Ta less than 0.2%, between 1 and 1.7% titanium,

[0153] less than 0.55% aluminium,

[0154] less than 0.5% cobalt

[0155] less than 0.03% carbon,

[0156] less than 0.35% manganese,

[0157] less than 0.2% silicon,

[0158] less than 0.006% boron,

[0159] less than 0.015% phosphorus,

[0160] less than 0.01% sulphur,

[0161] the residual percentage being iron and impurities resulting from processing.

[0162] In another particular case, the alloy is a nickel-base superalloy of 718PLUS type, with a weight content of:

[0163] between 12 and 20% chromium,

[0164] between 2 and 4% molybdenum,

[0165] at least one of the elements niobium or tantalum, such that the sum of niobium or tantalum is between 5 and 7%, with Ta lower than 0.2%,

[0166] between 1 and 2% tungsten,

[0167] between 5 and 10% cobalt,

[0168] between 0.4 and 1.4% titanium,

[0169] between 0.6 and 2.6% aluminium,

[0170] between 6 and 14% iron,

[0171] less than 0.1% carbon,

[0172] less than 0.015% boron,

[0173] less than 0.03% phosphorus

[0174] the residual percentage being nickel and impurities resulting from processing.

[0175] In general, the alloy is a nickel-base superalloy characterized by a content of niobium+tantalum higher than 2.5% and by the presence of an intergranular phase of  $\text{Ni}_3\text{Nb—Ta}$  type ( $\delta$  phase) at between 800° C. and 1050° C. and by the presence of an intragranular phase of  $\text{Ni}_3(\text{Al—Ti})-(\gamma')$  type and/or of  $\text{Ni}_3\text{Nb—Ta}$  ( $\gamma''$ ) type at between 600 and 800° C. For a nickel-base superalloy containing more than 2.5% niobium and/or tantalum and characterized by the presence of an intergranular phase containing niobium and/or tantalum and of  $\text{Ni}_3\text{Nb—Ta}$  type, the effect of the invention is also found even in the absence of the  $\gamma''$  hardening phase  $\text{Ni}_3\text{Nb—Ta}$ . The greater dissolution of the intergranular phase of delta  $\text{Ni}_3\text{Nb—Ta}$  type therefore releases niobium ( $\gamma'$ -gen element) which inserts itself in solid solution in the  $\gamma'$  hardening phase  $\text{—Ni}_3(\text{Al, Ti})$  and hardens the latter.

[0176] The treatment of the invention may comprise a fourth step allowing completion of the precipitation of the gamma" ( $\text{Ni}_3\text{Nb—Ta—}\gamma''$ ) and/or gamma' ( $\text{Ni}_3(\text{Al—Ti})-\gamma'$ ) hardening phases at a temperature lower than the temperature of the third step.

[0177] For example, provision can be made for a third step at between 700 and 750° C. for 4 h to 16 h followed by cooling at 50° C./h+/-10° C./h down to the temperature of the fourth step at between 600° C. and 650° C. which is held for between 4 h and 16 h.

[0178] The treatment of the invention may also comprise at least one intermediate step of short duration (no more than 1 h, see FIG. 2) between the first step and the second step to facilitate homogenisation of the temperature within parts of large-size during the temperature rise between the two first steps.

[0179] According to the invention, in which the (Ta+Nb) content of the alloy is at least 2.5%, it is recommended that the Al content does not exceed 3%, so as not to cause precipitation of the  $\gamma'$  phase at the grain boundaries. Over and above 3% Al, the  $\gamma'$  phase tends to stabilise to the detriment of the  $\delta$  phase and the Nb comes to insert itself in the  $\gamma'$  phase.



[0180] Also, still to give priority to the precipitation of the  $\delta$  phase at the grain boundaries, it is preferable that the (Nb+Ta+Ti)/Al ratio should be 3 or higher.

[0181] The invention will now be illustrated by several examples of embodiment of the heat treatment according to the invention, given in non-limiting manner.

[0182] The first examples of embodiment of the method according to the invention are applied to articles in 718 alloy obtained after thermo-mechanical treatment of an alloy obtained via conventional route of VIM+VAR+forging, but which could just as well have been obtained by powder metallurgy, and typically intended for the manufacture of aeronautic turbine discs.

[0183] On experimental level, using a VIM method we prepared then re-melted ingots in 718 alloy using the VAR method which were then hot-worked according to three different schedules of thermo-mechanical treatment (TTM, cf Table 2) numbered 1 to 3 in Table 2. The products obtained after thermo-mechanical treatment were cut up into samples (designated A to P in Table 1). The samples were then subjected to different heat treatments (TTH) comprising two to four steps depending on different cases (see Table 2).

[0184] The schedule for thermo-mechanical treatment N° 1 comprised rolling conducted with different passes at a temperature higher than the  $\delta$  phase solvus temperature of the alloy. The products formed according to thermo-mechanical treatment schedule N° 1 are bars whose metallurgical structure is devoid of delta phase (metallurgical state 2). In Table 2 the samples F, K, L, N were produced from bars obtained according to this first thermo-mechanical treatment schedule.

[0185] The thermo-mechanical treatment schedule N° 2 was a conventional forging schedule with reheat (by <<heat>> is meant holding in a furnace followed by deformation; <<reheat>> therefore means two deformation steps, each being preceded by holding in the furnace) at a temperature lower than the  $\delta$  phase solvus temperature of the alloy (<<sub-solvus temperature>>). This schedule allows precipitation of the  $\delta$  phase in the alloy. The products formed according to the thermo-mechanical treatment schedule N° 2 are so-called pancakes (a product globally in the rough shape of a flat disc resulting from deformation by forging) whose metallurgical structure contains some  $\delta$  phase distributed heterogeneously at the grain boundaries (metallurgical state 1, see FIGS. 4 and 5). In Table 2, the samples C, E and H were produced from pancakes obtained according to this second thermo-mechanical treatment schedule.

[0186] The thermo-mechanical treatment range N° 3 was a conventional stamping schedule in a single heat step at a temperature lower than the  $\delta$  phase solvus of the alloy. The products formed according to the thermo-mechanical treatment schedule N° 3 are blanks of discs whose metallurgical structure contains some  $\delta$  phase distributed in highly heterogeneous manner at the grain boundaries (Metallurgy state 1, see FIGS. 4 and 5). In Table 2, the samples A, B, D, G, I, J, M, O and P were prepared from blanks of turbine discs obtained according to this third thermo-mechanical treatment schedule.

[0187] Samples A to P were then subjected to five different schedules of heat treatment (<<TTH>>) designated a, b, c, d, e (TTH column, in Table 2) comprising two to four steps as per each case.

[0188] The heat treatment schedules of types <<a>> or <<b>> are reference heat treatment schedules representing the state of the art.

[0189] The heat treatment schedules of type <<a>> comprise one step of so-called isothermal solution treatment and two ageing steps. For these schedules, the solution heat treatment for samples A, B, C, D, F and P consisted of holding the alloy at a constant temperature of between 955 and 1010° C. for 40 to 90 minutes. The two ageing steps consisted of one hold at 720° C. for 8 hours followed by controlled cooling at 50° C./h down to a temperature hold of 620° C. for 8 hours.

[0190] The type <<b>> heat treatment schedule known as <<Direct Aged>> does not comprise any solution heat treatment and consists solely of two ageing steps conforming to type <<a>> treatments. Only sample E was subjected to the type <<b>> schedule.

[0191] The type <<c>> heat treatment schedules are in conformity with the invention and comprise two so-called solution heat treatment steps, respectively designated as the 1<sup>st</sup> step and 2<sup>nd</sup> step, and one or two ageing steps, respectively designated as the 3<sup>rd</sup> step and 4<sup>th</sup> step.

[0192] For these schedules which concerned samples G, H, J, K, M and N, the 1<sup>st</sup> solution heat treatment step consisted of holding the alloy at a constant temperature of between 940° C. and 980° C. for about 50 to 60 minutes. The 2nd solution treatment step consisted of holding the alloy at a constant temperature of between 980° C. and 1005° C. for about 15 to 40 minutes. The changeover from the 1<sup>st</sup> temperature hold to the 2<sup>nd</sup> temperature hold was performed by controlled reheating at a rate of about 2° C./min. The 3<sup>rd</sup> and 4<sup>th</sup> ageing steps were in conformity with the corresponding ageing steps of the type <<a>> reference schedules except for samples H and J.

[0193] With regard to sample H, the temperature of the 3<sup>rd</sup> ageing treatment step was brought to 750° C. instead of 720° C. as used for the other samples. This difference allowed the demonstration that the field of the invention is not limited to restricted temperature conditions and duration of ageing steps, but on the contrary that the invention can also be applied with temperatures and durations of ageing steps such as those used in the field of nickel-base superalloys.

[0194] As for sample J, this sample was only subjected to one ageing step at 720° C. for 10 hours. The ageing treatment applied to sample J shows that invention can also be applied when the alloy only undergoes a single ageing treatment step.

[0195] The type <<d>> schedules of heat treatments comprised two solution heat treatment steps and two ageing steps. Samples I and L were treated in accordance with these schedules. However these treatments do not conform to the invention on account of a second step conducted at a temperature that is too high or for duration that is too long. The conditions of the 2<sup>nd</sup> step effectively cause too extensive dissolution of the  $\delta$  phase and grain growth is no longer controlled, leading to major, uncontrolled grain enlargement during the second step for samples I and L.

[0196] The type <<e>> heat treatment schedule comprised a single solution heat treatment step at 1005° C. for 15 minutes, and two ageing steps. Only sample O was obtained according to this heat treatment schedule which does not conform to the invention as explained below.

[0197] Samples A to L and O were alloys of type 718 with 5.3% of Nb and 40 ppm of P. Sample N was an alloy of type 718 with 5.0% Nb and 40 ppm of P. Samples M and P were alloys of type 718 with 5.3% Nb and 80 ppm of P.



TABLE 1

compositions of tested samples										
Samples	Ni %	Fe %	Cr %	Al %	Ti %	Nb %	Mo %	B %	C %	P %
A-L, O	54.2	resid.	17.9	0.5	0.97	5.3	3	0.003	0.03	0.004
N	53.7	resid.	17.9	0.49	0.98	5.0	3	0.003	0.02	0.004
M, P	54.0	resid.	18.1	0.5	1.00	5.3	3	0.003	0.03	0.008

**[0198]** Table 2 summarizes the treatment conditions for the different samples, and the ASTM grain sizes and percentages of surface  $\delta$  phase which can be seen in a micrograph.

**[0199]** Table 3 summarizes the main mechanical properties of some of these samples, namely:

**[0200]** the yield strength (YS) during a tensile test at 20° C.

**[0201]** the ultimate tensile strength during a tensile test at 20° C. (UTS)

**[0202]** the number of cycles before rupture during a fatigue test at 450° C., comprising, with sinusoidal cycle and maximum stress of 1050 MPa, a frequency of 10 Hz and a load ratio R of 0.05;

**[0203]** the lifetime during a creep test at 650° C. under a stress of 550 MPa and under a stress of 690 MPa.

**[0204]** The grain size is defined according to the ASTM standard and if the grain size is relatively inhomogeneous, the maximum grain size (ALA) is also specified.

**[0207]** Product L was treated with two-step solution heat treatment but with a second step conducted at too high temperature and with too long duration, outside the field of the invention for a 718 alloy.

**[0208]** Products K and N do not have the same niobium content, but both were subjected to a heat treatment schedule of type <<c>> according to the invention.

**[0209]** The products in 718 alloy identified as C, E and H were transformed as per the thermo-mechanical schedule n° 2 which allows heterogeneous precipitation of the  $\delta$  phase.

**[0210]** Product C is a reference sample which, after the thermo-mechanical schedule n° 2, was treated as per standard <<a>> type heat treatment of alloy 718 (treatment comprising only one solution heat treatment at sub-solvus temperature).

**[0211]** Product E is also a reference sample which, after thermo-mechanical schedule n° 2, was treated as per type <<b>> heat treatment schedule and was therefore directly aged after forging and therefore did not undergo solution heat treatment before ageing.

TABLE 2

Characteristics and treatments of the different test samples																
					Solution heat treatment steps					Ageing steps						
					1 <sup>st</sup> step			2 <sup>nd</sup> step		3 <sup>rd</sup> step cooling			4 <sup>th</sup> step		Microstructure	
					T° (° C.)	Time (min.)	Ramp ° C./min	T° (° C.)	Time (min.)	T° (° C.)	Time (h.)	° C./h	T° (° C.)	Time (h.)	Grain ASTM	% δ
Sample	Alloy	Nb %	TTM	TTH	T° (° C.)	Time (min.)	° C./min	T° (° C.)	Time (min.)	T° (° C.)	Time (h.)	° C./h	T° (° C.)	Time (h.)	Grain ASTM	% δ
A	718	5.3	3	a	955	60	—	—	—	720	8	50	620	8	11-12	5.9
B	718	5.3	3.	a	970	60	—	—	—	720	8	50	620	8	11-12	5.1
C	718	5.3	2.	a	975	90	—	—	—	720	8	50	620	8	10	4.8
D	718	5.3	3.	a	1010	40	—	—	—	720	8	50	620	8	9	2
															ALA 5	
E	718	5.3	2.	b	—	—	—	—	—	720	8	50	620	8	10-14	3-5.5
F	718	5.3	1	a	970	60	—	—	—	720	8	50	620	8	9-10	5.5
G	718	5.3	3	c	980	60	2	1005	15	720	8	50	620	8	11-12	3.1
H	718	5.3	2	c	980	60	2	1005	15	750	8	50	620	8	10	2.9
I	718	5.3	3	d	970	80	2	1005	60	720	8	50	620	8	9	1.9
															ALA 5	
J	718	5.3	3	c	970	50	2	995	40	720	10	—	—	—	11-12	3.5
K	718	5.3	1	c	980	60	2	1000	20	720	8	50	620	8	9-10	3.3
L	718	5.3	1	d	975	60	2	1015	20	720	8	50	620	8	8-9	1.4
															ALA 4	
M	718	5.3	3	c	980	60	2	1005	15	720	8	50	620	8	11-12	3.0
N	718	5.0	1	c	940	60	2	980	15	720	8	50	620	8	9-10	3.4
O	718	5.3	3	e				1005	15	720	8	50	620	8	10	3.2
															ALA 5	
P	718	5.3	3	a	970	60	—	—	—	720	8	50	620	8	11-12	5.3

**[0205]** The products in 718 alloy, F, K, L, N were therefore transformed according to thermo-mechanical schedule n° 1 which does not allow  $\delta$  phase precipitation.

**[0206]** Product F is a reference sample which, after thermo-mechanical treatment schedule n° 1, was treated using standard type <<a>> thermo-mechanical treatment of alloy 718 (treatment comprising a single solution heat treatment step at  $\delta$  phase sub-solvus).

**[0212]** Product H was subjected to heat treatment according to the invention (type <<c>>) with two-step solution heat treatment within the field of the invention.

**[0213]** The products in 718 alloy identified as A, B, D, G, I, J, M, O and P were transformed according to thermo-mechanical schedule n° 3 which allows highly heterogeneous precipitation of the  $\delta$  phase.



[0214] After thermo-mechanical treatment n° 3, the products A, B and P were treated as per the standard treatment for alloy 718 (treatment of type <<a>> comprising a single sub-solvus solution heat treatment).

[0215] Product D was treated with treatment comprising only one solution heat treatment step but at higher temperature than for products A, B and P, i.e. at a temperature close to  $\delta$  phase solvus.

[0216] After thermo-mechanical treatment, product I was subjected to two-step solution heat treatment but whose duration for the second step was too long having regard to the temperature. The heat treatment applied to product I therefore lies outside the field of the invention.

[0217] After thermo-mechanical treatment n° 3, product G was treated with two-step solution heat treatment within the field of the invention (heat treatment <<c>>). Product J was also treated with two-step solution heat treatment within the field of the invention, but was not treated with a fourth step.

[0218] Product M was treated with two-step solution heat treatment within the field of the invention, but has a phosphorus content of 0.008% which is twice higher than that of products A-L and N-O.

[0219] Product O was subjected to <<e>> heat treatment with one-step solution heat treatment; this treatment lies outside the field of the invention.

[0220] Product P is a reference sample having a phosphorus content of 0.008%. It was treated using a standard treatment schedule for alloy 718 (treatment of <<a>> type comprising a only one solution heat treatment at sub-solvus temperature).

[0221] Products A, B, C which were treated with standard sub-solvus heat treatment (schedule type <<a>>) have a fine grain microstructure (>9 ASTM) but comprise a fraction of  $\delta$  phase (>4.5%) greater than the preferably desired  $\delta$  phase fraction according to the invention. The mechanical properties obtained with these products constitute the reference for assessment of the tensile, fatigue and creep properties obtained with the thermo-mechanical schedules (TTM) 2 and 3.

TABLE 3

Mechanical properties of the tested samples					
Sample	Tensile 20° C.		Fatigue 450° C. R = 0.05 10 Hz $\sigma_{max}$ =	Creep lifetime (h)	
	YS (MPa)	UTS (MPa)	1050 MPa	650° C.	
			Lifetime (cycles)	550 MPa	690 MPa
A	1210	1470	>3 000 000	290	40
B	1240	1480	>3 500 000	340	60
C	1170	1465	1 400 000	350	70
D	1310	1495	60 000	1600	200
E	1350	1520	>3 000 000	180	40
G	1340	1520	>3 000 000	940	120
H	1290	1505	>3 000 000	770	150
M	1335	1520	>3 000 000	1400	330
P	1245	1492	>3 000 000	500	80

[0222] Product D was treated at a higher temperature than products A, B, and C, it comprises ASTM 5 size grains and a  $\delta$  phase distributed heterogeneously (<2.5%) that is lower than the preferably desired  $\delta$  phase according to the invention. It is ascertained that this treatment did not allow a fine grain microstructure to be maintained (at least ASTM 7, preferably at least 8, and best ASTM 9) nor the satisfactory fatigue

properties found for products A, B, and C. The considerable reduction in the fatigue lifetimes is attributable to the presence of large-size ASTM 5 grains which form fatigue initiator sites.

[0223] Product E which was directly aged after thermo-mechanical treatment N° 2 has a very heterogeneous grain size (ASTM 10 to 14) and major variations in the level of  $\delta$  phase, this level being found in most of the regions of the part (in particular the regions subject to creep) and is higher than the desired fraction of  $\delta$  phase. Although the tensile and fatigue properties of product E are greater than those of products A, B, C, it is ascertained that the creep lifetimes obtained with product E are shorter than the creep lifetimes for products A, B, C.

[0224] The absence of solution heat treatment does not allow homogenization of the microstructure and is responsible for the presence of very fine grains (>12 ASTM) and of  $\delta$  phase fractions that are too large, which are the cause of this degradation of the creep properties.

[0225] The absence of solution heat treatment for product E also allows the maintaining of residual work hardening after forging, which is beneficial for tensile properties but is detrimental to creep resistance in the low stress area.

[0226] Products G, H, M were treated within the field of the invention and have a fine grain microstructure (>9 ASTM) and a  $\delta$  phase fraction (2.9% and 3.5%) included within the desired range of  $\delta$  phase fraction, namely no more than 4% and at least 2.5%. It is ascertained that the tensile properties are distinctly greater than those of products A, B, C and of the same level as those of product E. It is also ascertained that the creep properties of products G, H, M are distinctly better than those of products A, B, C, E whereas the grain size is similar in these products. The fine grain microstructure of products G, H, M allows preservation of the fatigue properties obtained with products A, B, C, E, and the smaller  $\delta$  phase fraction of products G, H, M allows for improved creep resistance.

[0227] The comparison between samples B and P shows that the increase in phosphorus content for a 718 alloy subjected to a reference treatment (a) does not substantially improve creep resistance.

[0228] Unexpectedly, the application of a treatment according to the invention to product M which has a higher phosphorus content (80 ppm), allowed a considerable increase in creep lifetimes up to a factor of 4 compared with products A, B, C, and also compared with product P whose phosphorus content is comparable with that of product M but which was not given treatment according to the invention.

[0229] The combination of added phosphorus with the treatment of the invention therefore has a synergic effect which is positive on the creep properties of the alloy obtained.

[0230] The invention targets the maintaining of a residual  $\delta$  phase fraction (preferably higher than 2.5%) which allows satisfactory ductility to be maintained at high temperature. If the content of the  $\delta$  phase is too low this has an effect on damage and ductility under tensile testing at high temperature (650° C. with a strain rate of  $10^{-5}s^{-1}$ ). It is effectively ascertained that product D whose  $\delta$  phase content is close to 2% has a ductility (elongation at break of 7%) that is much lower than that of product G (elongation at break of 27%) whose  $\delta$  phase fraction is close to 3%. This reduced ductility of product D results from intergranular damage caused by a fraction of  $\delta$  phase that is too small and distributed heterogeneously.

[0231] The influence of the treatments of the invention on the microstructure will now be detailed.



[0232] An examination was made of samples A, B, C, D, E, G, H, I, J, M, O and P which are in 718 alloy and were transformed using thermo-mechanical schedule N° 2 or n° 3.

[0233] FIGS. 4 to 9 are micrographs representing the microstructures:

[0234] of samples A, B, C, D, E, G, H, I, J, M, O and P in their initial state after thermo-mechanical treatment (FIGS. 4 and 5),

[0235] of samples D and O after being subjected to heat treatment comprising only one solution heat treatment step (FIGS. 6 and 7)

[0236] of samples G, H and M after being subjected to heat treatment according to the invention (FIGS. 8 and 9).

[0237] FIGS. 4 and 5 illustrate the microstructure of samples A, B, C, D, E, G, H, I, J, M, O and P (metallurgical state 1) after being subjected to sub-solvus thermo-mechanical deformation (thermo-mechanical schedule 2 or 3). This is a microstructure which shows delta phase  $\text{Ni}_3\text{Nb}-\delta$  and/or  $\text{Ni}_3\text{Ta}-\delta$  at the grain boundaries, but in a manner not uniformly distributed between the grains.

[0238] FIG. 4 shows that the samples have a fine grain of about size ASTM 11, with heterogeneous distribution of the  $\delta$  phase (black spots at the grain boundaries). After the thermo-mechanical deformation schedule, the  $\delta$  phase percentage is 2.8 to 6% and the grain size is ASTM 10 to 13. This gives a very heterogeneous microstructure from these two viewpoints.

[0239] FIG. 5 illustrates the microstructure of the samples with greater enlargement and shows grains whose boundaries are mostly fully devoid of  $\delta$  phase (this phase is shown in white in this micrograph).

[0240] When a treatment is applied to a sample (sample B) that only comprises one solution heat treatment step at 970° C. for about 60 minutes, the percentage of  $\delta$  phase obtained is 4.7 to 5.5% and the grain size of ASTM 11 to 12. The homogeneity of the sample is therefore improved but a large fraction of  $\delta$  phase is maintained, for which it is known (see sample B, Tables 1 & 2) that is highly unfavourable for creep resistance.

[0241] If the heat treatment applied to a sample (see for example sample O in Table 1) is treatment only comprising one solution heat treatment step at 1005° C. for about 15 minutes, corresponding to the <<second step>> of the invention, the percentage of  $\delta$  phase obtained (see FIGS. 6 and 7) is 1.1 to 3.5%, and the grain size is ASTM 5 to 9. The level of  $\delta$  phase is therefore reduced, which is in the right direction for creep resistance, but heterogeneous distribution of grain size is observed. This can be accounted for by the heterogeneous grain growth during this step resulting from non-homogeneous distribution of the  $\delta$  phase inherited from the initial microstructure.

[0242] Indeed, and as previously explained, the initial microstructure results from sub-solvus deformation (state 1), the distribution of the  $\delta$  phase is heterogeneous in the initial microstructure. As a result, some grains may contain a large quantity of  $\delta$  phase at the grain boundaries in the initial structure, whereas other grains only have little or no  $\delta$  phase at the grain boundaries (see FIG. 5).

[0243] By conducting heat treatment directly at the temperature of the second step, without any intermediate temperature hold at the temperature of the first step in accordance with the invention, the grains which are not surrounded with  $\delta$  phase or which have little  $\delta$  phase at the grain boundaries will enlarge uncontrollably up to a grain size which may exceed about ASTM 5-6, whereas the growth of the other grains surrounded by  $\delta$  phase will be hindered and will give

rise to grain sizes close to ASTM 9. This heterogeneity of grain size can be clearly seen in the micrographs in FIGS. 6 and 7. The presence, even much localized presence of ASTM 5-6 grains considerably reduces the fatigue lifetimes.

[0244] On the other hand, if some samples (samples G, H and M) are given the heat treatment according to the invention, namely a first step at 980° C. for 60 min and, immediately afterwards, heating as per a ramp of 2° C./min up to a second temperature hold at 1005° C. for 15 min, a  $\delta$  phase of 2.9 to 3.5% is obtained with a grain size of ASTM 10 to 12.

[0245] The micrographs in FIGS. 8 and 9 show that, compared with the initial state of the sample:

[0246] the grain size is more homogeneous and remains very fine;

[0247] the  $\delta$  phase is now distributed regularly at the grain boundaries, which efficiently prevents the growth thereof.

[0248] By means of the small formation of  $\delta$  phase precipitates, which leaves the Nb and Ta elements available in dissolved form, by means of the reduced grain size, of the homogeneous distribution of the  $\delta$  phase at the grain boundaries and of the well-adjusted level of the presence of this  $\delta$  phase, creep resistance and tensile strength are improved. It is in particular the fine grain size associated with controlled dissolution of the  $\delta$  phase which allows the objectives of the invention to be reached, which are:

[0249] strong fatigue properties, avoiding premature crack initiation in large grains and giving priority to crack initiation in niobium carbides;

[0250] improved yield strength due to more extensive hardening generated by a larger fraction of hardening phase;

[0251] a distinct, even considerable, improvement in the creep resistance of the alloy with sufficient phosphorus content (sample M).

[0252] Once the alloy has been treated according to the invention, the finishing operations can proceed as is usual in the prior art to obtain the end part.

[0253] In addition, the inventors conducted additional tests on samples of alloys of type 718Plus and 725, and were able to confirm that the invention when applied to other nickel-base superalloys having a niobium and/or tantalum content of more than 2.5%, allowed a marked improvement in their creep resistance and tensile strength.

1. A method for manufacturing a blank of a part in Ni-base superalloy comprising at least 50 Ni in weight percent, comprising:

preparing an alloy of said superalloy, and

conducting heat treatments of said alloy, wherein:

said superalloy in weight percentage comprises at least a total of 2.5% Nb and Ta;

heat treatment is applied to said alloy, comprising a plurality of steps distributed as follows:

in a first step, during said alloy is held at between 850 and 1000° C. for at least 20 minutes, precipitating a  $\delta$  phase at grain boundaries;

in a second step, during said alloy is held at a temperature higher than the temperature of the first step, allowing partial dissolution of the  $\delta$  phase obtained at the first step, and after the second step to obtain a  $\delta$  phase quantity of between 2 and 4%, the first and second step being conducted without intermediate cooling; and

in ageing treatment comprising a third step and optionally one or more additional steps conducted



- at a temperature lower than that of the first step, allowing precipitation of  $\gamma'$  and/or  $\gamma''$  hardening phases;  
the first step being conducted between 900 and 1000° C. for at least 30 min, and the second step at between 940 and 1020° C. for 5 to 90 min, the difference in temperature between the two steps being at least 20° C.
2. The method according to claim 1, wherein an Al content of the alloy is equal to or less than 3%.
3. The method according to claim 1, wherein a ratio (Nb+Ta+Ti)/Al of the alloy is equal to or more than 3.
4. The method according to claim 1, wherein a grain size obtained at the end of the alloy treatment is between 7 and 13 ASTM.
5. The method according to claim 1, wherein distribution of the  $\delta$  phase is homogeneous at the grain boundaries after the ageing treatment.
6. The method according to claim 1, wherein after the second step a  $\delta$  phase quantity is obtained of between 2.5 and 3.5%.
7. (canceled)
8. The process according to claim 1, wherein a changeover from the first step to the second step is performed at a rate of 4° C./min or less.
9. (canceled)
10. The process according to claim 1, wherein the alloy comprises by weight:  
between 50 and 55% nickel,  
between 17 and 21% chromium,  
less than 0.08% carbon,  
less than 0.35% manganese,  
less than 0.35% silicon,  
less than 1% cobalt  
between 2.8 and 3.3% molybdenum,  
at least one of the elements niobium or tantalum, such that the sum of niobium and tantalum totals between 4.75 and 5.5% with Ta less than 0.2%,  
between 0.65 and 1.15% titanium,  
between 0.20 and 0.80% aluminium,  
less than 0.006% boron,  
less than 0.015% phosphorus,  
the residual percentage being iron and impurities resulting from processing.
11. The method according to claim 10 wherein the first step is conducted at between 920 and 990° C. for at least 30 min and the second step is conducted at a temperature of between 960 and 1010° C. for 5 to 45 min.
12. The method according to claim 11, wherein the total content of Nb and Ta of the alloy is between 5.2 and 5.5%, in that the first step is conducted at between 960 and 990° C. for 45 min to 2 h, and in that the second step is conducted at between 990 and 1010° C. for 5 to 45 min.
13. The method according to claim 11, wherein a total content of Nb and Ta of the alloy is between 4.8 and 5.2%, in that the first step is conducted at between 920 and 960° C. for 45 min to 2 h, and in that the second step is conducted at between 960 and 990° C. for 5 to 45 min.
14. The method according to claim 1, wherein the alloy comprises a weight content of:  
between 55 and 61% nickel,  
between 19 and 22.5% chromium,  
between 7 and 9.5% molybdenum,

- at least one of the elements niobium or tantalum, such that the sum of niobium and tantalum is between 2.75 and 4% with Ta less than 0.2%,  
between 1 and 1.7% titanium,  
less than 0.55% aluminium,  
less than 0.5% cobalt,  
less than 0.03% carbon,  
less than 0.35% manganese,  
less than 0.2% silicon,  
less than 0.006% boron,  
less than 0.015% phosphorus,  
less than 0.01% sulphur,  
the residual percentage being iron and impurities resulting from processing.
15. The method according to claim 1, wherein the alloy comprises by weight:  
between 12 and 20% chromium,  
between 2 and 4% molybdenum,  
at least one of the elements niobium or tantalum, such that the sum of niobium or tantalum is between 5 and 7% with Ta less than 0.2%,  
between 1 and 2% tungsten,  
between 5 and 10% cobalt,  
between 0.4 and 1.4% titanium,  
between 0.6 and 2.6% aluminium,  
between 6 and 14% iron,  
less than 0.1% carbon,  
less than 0.015% boron,  
less than 0.03% phosphorus  
the residual percentage being nickel and impurities resulting from processing.
16. The method according to claim 1, wherein the alloy has a weight percent content of phosphorus of more than 0.007%.
17. The method according to claim 1, wherein the first step and the second step are conducted at sub-solvus temperatures of the  $\delta$  phase of the alloy, the first step being conducted at a temperature between 50° C. below the  $\delta$  solvus temperature and 20° C. below the  $\delta$  solvus temperature, and the second step being conducted at a temperature between 20° C. below the  $\delta$  solvus temperature and the  $\delta$  solvus temperature.
18. The method according to claim 1, wherein a temperature of the hot-worked blank part is held constant during at least one of the said steps.
19. The method according to claim 1, wherein the said third step is conducted at between 700 and 750° C. for 4 to 16 h and in that a fourth step is conducted at between 600 and 650° C. for between 4 and 16 h, cooling at 50° C./h to  $\pm 10^\circ$  C./h being carried out between the said third and fourth steps.
20. The method according to claim 1, wherein between the first and second steps, the hot-worked alloy is held at least at one intermediate temperature between the temperatures of the first and second steps for no more than 1 h.
21. The method according to claim 1, wherein said blank part was prepared in ingot form and then hot-worked.
22. The method according to claim 1, wherein said blank part was prepared using a powder metallurgy method.
23. A part in nickel-base superalloy wherein it is obtained from a blank part manufactured using the method according to claim 1.
24. The part according to claim 23, wherein it is an aeronautic or land-based gas turbine part.