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(54) **Method of controlling and refining final grain size in supersolvus heat treated nickel-base superalloys**

(57) A method of forming a component from a gamma prime precipitation-strengthened nickel-base superalloy. The method entails formulating the superalloy to have a sufficiently high carbon content and forging the superalloy at sufficiently high local strain rates so that,

following a supersolvus heat treatment, the component is characterized by a fine and substantially uniform grain size distribution, preferably finer than ASTM 7 and more preferably in a range of about ASTM 8 to 10.

EP 2 019 150 A1

Description

BACKGROUND OF THE INVENTION

5 **[0001]** The present invention generally relates to methods for processing nickel-base superalloys. More particularly, this invention relates to a method of forging an article from a nickel-base superalloy, in which increased local strain rates in combination with increased carbon content promote a more controlled grain growth during supersolvus heat treatment, such that the article is characterized by a microstructure with a finer uniform grain size.

10 **[0002]** Gamma prime (γ') precipitation-strengthened nickel-base superalloys contain chromium, tungsten, molybdenum, rhenium and/or cobalt as principal elements that combine with nickel to form the gamma (γ) matrix, and contain aluminum, titanium, tantalum, niobium, and/or vanadium as principal elements that combine with nickel to form the desirable gamma prime precipitate strengthening phase, principally $\text{Ni}_3(\text{Al}, \text{Ti})$. Gamma prime precipitation-strengthened nickel-base superalloys (hereinafter, gamma prime nickel-base superalloys) are widely used for disks and other critical gas turbine engine components forged from billets produced by powder metallurgy (P/M), conventional cast and wrought processing, and spraycast or nucleated casting forming techniques. Gamma prime nickel-base superalloys formed by powder metallurgy are particularly capable of providing a good balance of creep, tensile, and fatigue crack growth properties to meet the performance requirements of certain gas turbine engine components such as turbine disks. In a typical powder metallurgy process, a powder of the desired superalloy undergoes consolidation, such as by hot isostatic pressing (HIP) and/or extrusion consolidation. The resulting billet is then isothermally forged at temperatures slightly below the gamma prime solvus temperature of the alloy to approach superplastic forming conditions, which allows the filling of the die cavity through the accumulation of high geometric strains without the accumulation of significant metallurgical strains. These processing steps are designed to retain the fine grain size originally within the billet (for example, ASTM 10 to 13 or finer), achieve high plasticity to fill near-net-shape forging dies, avoid fracture during forging, and maintain relatively low forging and die stresses. (Reference throughout to ASTM grain sizes is in accordance with the scale established in ASTM Standard E 112.) In order to improve fatigue crack growth resistance and mechanical properties at elevated temperatures, these alloys are then heat treated above their gamma prime solvus temperature (generally referred to as supersolvus heat treatment), to cause significant, uniform coarsening of the grains.

25 **[0003]** Forged gas turbine engine components often contain grains with sizes of about ASTM 9 and coarser, such as ASTM 2 to 9, though a much tighter range is typically preferred, such as grain sizes within a limited range of 2 to 3 ASTM units. Such a limited range can be considered uniform, which as used herein refers to grain size and growth characterized by the substantial absence of nonuniform critical grain growth. As used herein, critical grain growth (CGG) refers to localized excessive grain growth in an alloy that results in the formation of grains outside typical uniform grain size distributions whose size sufficiently exceeds the average grain size in the alloy (such as regions as coarse as ASTM 00 in a field of ASTM 6-10) to negatively affect the low cycle fatigue (LCF) properties of an article formed from the alloy, manifested by early preferential crack nucleation in the CGG regions. Critical grain growth can also have a negative impact on other mechanical properties, such as tensile strength. Critical grain growth occurs during supersolvus heat treatment following hot forging operations in which a wide range of local strains and strain rates are introduced into the material. Though not wishing to be held to any particular theory, critical grain growth is believed to be driven by excessive stored energy within the worked article, and may involve individual grains, multiple individual grains within a small region, or large areas of adjacent grains. The grain diameters of the effected grains are often substantially coarser than the desired grain size. Disks and other critical gas turbine engine components forged from billets produced by powder metallurgy and extrusion consolidation have appeared to exhibit a lesser propensity for critical grain growth than if forged from billets produced by conventional cast and wrought processing or spraycast forming techniques, but in any event are susceptible to critical grain growth during supersolvus heat treatment.

40 **[0004]** Commonly-assigned U.S. Patent No. 4,957,567 to Krueger et al. teaches a process for eliminating critical (abnormal) grain growth in fine grain gamma-prime nickel-base superalloy components by controlling the localized strain rates experienced during the hot forging operation. Strain rate is defined as the instantaneous rate of change of geometric strain with time. Krueger et al. teach that local strain rates must generally remain below a critical value, ϵ_c , in order to avoid detrimental critical grain growth during subsequent supersolvus heat treatment. According to Krueger et al., the maximum strain rate is composition, microstructure, and temperature dependent, and can be determined for a given superalloy by deforming test samples under various strain rate conditions, followed by a suitable supersolvus heat treatment. The maximum (critical) strain rate is then defined as the strain rate that, if exceeded during deformation and working of a superalloy and accompanied by a sufficient amount of total strain, will result in critical grain growth after supersolvus heat treatment.

55 **[0005]** Another processing limitation identified by Krueger et al. as avoiding critical grain growth in a nickel-base superalloy having a gamma prime content of, for example, 30-46 volume percent and higher, is to ensure superplastic deformation of the billet during forging. For this purpose, the billet is processed to have a fine grain microstructure that achieves a minimum strain rate sensitivity (m) of about 0.3 or greater for the superalloy within the forging temperature

range. As known in the art, the ability of a fine grain billet to deform superplastically is dependent on strain rate sensitivity, and superplastic materials exhibit a low flow stress as represented by the following equation:

$$\sigma = K\dot{\epsilon}^m$$

where σ is the flow stress, K is a constant, $\dot{\epsilon}$ is the strain rate, and m is the strain rate sensitivity, with higher values of m corresponding to greater superplasticity.

[0006] Further improvements in the control of final grain size have been achieved with the teachings of commonly-assigned U.S. Patent No. 5,529,643 to Yoon et al. and U.S. Patent No. 5,584,947 to Raymond et al. In addition to the requirement for superplasticity during forging (in other words, maintaining a high m value), Raymond et al. teach the importance of a maximum strain rate in combination with chemistry control, particularly the carbon and/or yttrium content of the alloy to achieve grain boundary pinning in alloys having a gamma prime content of up to 65 volume percent. In a particular example, Raymond et al. cites an upper limit strain rate of below about 0.032 per second (s^{-1}) for a gamma prime nickel-base superalloy identified as Alloy D and commercially known as René 88DT (R88DT; U.S. Patent No. 4,957,567). In addition to maintaining a high m value, Yoon et al. also identifies a maximum strain rate of not more than about 0.032 s^{-1} , particularly in reference to forging an alloy identified in Yoon et al. as Alloy A, which again is R88DT. Yoon et al. further place an upper limit on the maximum strain rate gradient during forging, and requires extended annealing of the forging at a subsolvus temperature to remove stored strain energy prior to performing a supersolvus heat treatment. Finally, Yoon et al. achieve optimum superplasticity by forming the billet to have a grain size of finer than about ASTM 12, and maintaining the billet microstructure to achieve a minimum strain rate sensitivity of about $m=0.3$ within the forging temperature range.

[0007] While the teachings of Krueger et al., Yoon et al., and Raymond et al. have been largely effective in controlling critical grain growth, implementation of their teachings has generally required the use of very slow ram speed control of the forging press head (generally with a simple linear decay versus stroke control scheme), coupled by simulative modeling to translate the press head deformation rate into actual metal strain rate as a function of temperature, constitutive property data for the forging stock, die shape, and die or mult lubrication.

[0008] In addition to the absence of critical grain growth, mechanical properties of components forged from fine grain nickel-base superalloys further benefit from improved control of the grain size distribution to achieve a distribution and average grain size that are, respectively, as narrow and fine as possible. Such a capability is particularly beneficial for high temperature, high gamma prime content (e.g., about 30 volume percent and above) superalloys, such as R88DT, for which a desired uniform grain size is generally not coarser than ASTM 6 for gas turbine disks. Though prior forging practices of the type described above have achieved grain sizes in a range of ASTM 5 to 8, less than optimal mechanical properties can still result. For example, low cycle fatigue life is known to decrease with coarser average grain sizes, even if uniform. The impact of average grain size on low cycle fatigue properties of supersolvus heat treated P/M superalloys is most apparent at low to intermediate temperatures, such as in a range of about 400°F to about 750°F (about 200°C to about 400°C). While the overall temperature capability and balance of properties that P/M alloys offer are very attractive and relied on for the most advanced current engine applications, even more benefit from these alloys could be obtained if their low cycle fatigue properties at low to intermediate temperatures could be improved.

[0009] In view of the above, it would be desirable if more grain size refinement (finer average grain size) could be achieved in gas turbine engine forgings, along with the avoidance of localized critical grain growth. It would be particularly desirable if both of these goals could be achieved using a process whose parameters are within practical limits of manufacturability and cost constraints.

BRIEF SUMMARY OF THE INVENTION

[0010] The present invention provides a method of forming components from gamma prime nickel-base superalloys. The method entails formulating such a superalloy to have a sufficiently high carbon content and forging the superalloy at sufficiently high local strain rates so that, following a supersolvus heat treatment, the component is characterized by a fine and substantially uniform grain size distribution, preferably an average grain size finer than ASTM 7 and more preferably in an average range of about ASTM 8 to 10. The present invention is further capable of avoiding critical grain growth that would produce individual grains or small regions of grains having grain sizes of more than five and preferably three ASTM units coarser than the average grain size in the component, or large regions that are uniform in grain size but with a grain size coarser than a desired grain size range of about two ASTM units.

[0011] The method includes formulating the superalloy to have a composition suitable for producing forged polycrystalline articles subjected to high temperatures and dynamic loads, a notable example of which is a turbine disk of a gas turbine engine. An exemplary example of such an alloy is the aforementioned gamma prime precipitation-strengthened

nickel-base superalloy R88DT, though it is foreseeable that the teachings of this invention could be extended to other gamma prime nickel-base superalloys approximating the mechanical properties of R88DT critical to a turbine disk, such as low cycle fatigue life. In contrast to R88DT, the superalloy employed in the method of this invention is formulated to have a carbon content of at least 0.045 weight percent, and preferably in excess of 0.060 weight percent, which is the conventional upper limit for carbon in R88DT. A billet is formed of the superalloy and worked at a temperature below the gamma prime solvus temperature of the superalloy so as to form a worked article. In particular, the billet is worked while maintaining strain rates as high as possible to control average grain size, but below an upper strain rate limit to avoid critical grain growth. According to one aspect of the invention, the billet is not required to be worked superplastically, i.e., can have a strain rate sensitivity (m) of less than 0.3 at the working (e.g., forging) temperature. In fact, it is preferred to work the billet non-superplastically to achieve the finest grain sizes. The worked article is then heat treated at a temperature above the gamma prime solvus temperature of the superalloy for a duration sufficient to uniformly coarsen the grains of the worked article, after which the worked article is cooled at a rate sufficient to reprecipitate gamma prime within the worked article. The cooled worked article has an average grain size of not coarser than ASTM 6 and preferably not coarser than 7 ASTM, for example, in a range of about ASTM 8 to 10.

[0012] A significant advantage of this invention is that, in addition to avoiding critical grain growth and avoiding the necessity to forge superplastically, the higher strain rate limit of the process window for working the billet has been shown to achieve significant control of the average grain size in the component and achieve a uniform grain size distribution within a desired narrower range that is significantly finer than previously possible. In this manner, mechanical properties of the component, including low cycle fatigue and tensile strength, can be improved. Though not wishing to be held to any particular theory, it is believed that formulating a superalloy such as R88DT to contain a carbon level above its conventional upper limit (0.060 weight percent) allows the use of strain rates beyond the upper strain rate limit of 0.010 per second (s^{-1}) typically associated with R88DT, and even above the upper strain rate limit of 0.032 s^{-1} previously permitted by Yoon et al. and Raymond et al. for R88DT, resulting in components capable of exhibiting a more refined average grain size and substantially free of critical grain growth, which together improve the low cycle fatigue life of the component. Low cycle fatigue life is particularly improved within a temperature range of about 400°F to about 750°F (about 200°C to about 400°C) relative to R88DT with a conventional carbon content of up to 0.060 weight percent.

[0013] Improvements in low cycle fatigue life are believed possible, with the further benefit of higher temperature properties achieved with powder metallurgy alloys such as R88DT. Other benefits of the finer average grain size achieved with this invention include improved sonic inspection capability due to lower sonic noise, and improved yield behavior in service due to improved yield strength with finer grain size.

[0014] Other objects and advantages of this invention will be better appreciated from the following detailed description.

BRIEF DESCRIPTION OF THE DRAWINGS

[0015]

Figure 1 is a schematic graph representing strain rate versus temperature and resulting grain size distribution for conventional R88DT specimens and R88DT specimens modified to contain carbon levels in excess of 0.060 weight percent.

Figures 2 and 3 are graphs plotting average and ALA grain size, respectively, for R88DT specimens modified to contain either 0.066 or 0.070 weight percent carbon and processed with relatively fast (at least 0.01 s^{-1}) and slow (less than 0.01 s^{-1}) strain rate regimes.

Figure 4 is a graph plotting strain rate sensitivity value and average grain size versus strain rate for data of Figure 2 corresponding to specimens forged at 1875°F.

Figure 5 is a graph plotting the average and ALA grain size versus forging temperature data in Figures 2 and 3 for the R88DT specimens containing 0.070 weight percent carbon.

Figures 6 and 7 are normal probability plots of the average and ALA grain size, respectively, for specimens formulated and forged in accordance with current practices and the present invention.

Figure 8 is a bar graph comparing the sonicability of the forged specimens of Figures 6 and 7.

Figures 9 and 10 are normal probability plots of the ultimate tensile strength and yield strength, size, respectively, of the forged specimens of Figures 6 and 7.

DETAILED DESCRIPTION OF THE INVENTION

[0016] The present invention is particularly directed to components formed by forging gamma prime precipitation-strengthened nickel-base superalloys. A particular example is high pressure turbine disks of gas turbine engines, which are typically formed by isothermally forging a fine-grained billet at temperatures at or near the recrystallization temperature of the alloy but less than the gamma prime solvus temperature of the alloy, and under superplastic forming conditions to enable filling of the forging die cavity through the accumulation of high geometric strains without the accumulation of significant metallurgical strains. After forging, a supersolvus heat treatment is performed, during which grain growth occurs. In the past, such a supersolvus heat treatment has typically yielded an acceptable but not wholly optimal average grain size range of about ASTM 2 to 9. In accordance with commonly-assigned U.S. Patent Nos. 4,957,567 to Krueger et al., 5,529,643 to Yoon et al., and 5,584,947 to Raymond et al., whose teachings regarding strain rates, strain rate gradients, and superplasticity are incorporated herein by reference, placing an upper limit on the strain rate (critical strain rate), an upper limit on the strain rate gradient (critical strain rate gradient), and a strain rate sensitivity (m) of at least about 0.3 during forging avoids critical grain growth during supersolvus heat treatment. However, even with the benefits of Krueger et al., Yoon et al., and Raymond et al., grain sizes in forged gamma prime precipitation-strengthened nickel-base superalloys have typically been limited to a range of about ASTM 5 to 8.

[0017] The present invention identifies processing parameters by which a finer average grain size and more desirable grain size distribution can be achieved in a gamma prime precipitation-strengthened nickel-base superalloy, in addition to avoidance of critical grain growth. According to one aspect of the invention, a finer and more controllable average grain size can be achieved by increasing the strain rate during forging, resulting in a strain rate window having an upper limit that is higher than conventionally believed possible without inducing critical grain growth. The upper limit of the strain rate window corresponds to the maximum strain rate at which critical grain growth can be avoided. According to a second aspect of the invention, higher strain rates and non-superplastic deformation can be employed without causing critical grain growth by modifying an alloy to have a relatively high carbon content.

[0018] The above-noted aspects of the invention will be discussed in reference to processing of a high-pressure turbine disk for a gas turbine engine. However, those skilled in the art will appreciate that the teachings and benefits of this invention are applicable to numerous other components that undergo forging and in which a fine controlled grain size distribution is desired.

[0019] In the production of a high pressure turbine disk from a gamma prime nickel-base superalloy, a billet is typically formed by powder metallurgy (P/M), a cast and wrought processing, or a spraycast or nucleated casting type technique. Such processes are carried out to yield a billet with a fine grain size, typically about ASTM 10 or finer, to achieve low flow stresses during forging. As previously noted, the ability of a fine grain billet to deform superplastically is dependent on strain rate sensitivity (m). Whether formed by powder metallurgy, spraycast forming, cast and wrought, or another suitable method, prior art billets for high pressure turbine disks have been formed under conditions, including a specified temperature range, to produce the desired fine grain size and also maintain a minimum strain rate sensitivity (m) of about 0.3 or greater within the forging temperature range. Alternatively, to control the strain rate sensitivity, it has been conventional practice to control the forging process to be superplastic by forging in a regime of strain rate and temperature where flow stress is constant for any strain (negligible strain hardening or strain softening). As will be discussed in more detail below, with the present invention it is believed that strain rates higher than previously thought possible can be employed to produce suitable forgings without the forging process being fully superplastic, i.e., at strain rate sensitivity values of less than about 0.3.

[0020] In a preferred embodiment utilizing a powder metallurgy process, the billet can be formed by consolidating a superalloy powder, such as by hot isostatic pressing (HIP) or extrusion consolidation, the latter of which preferably uses a sufficiently low ram speed to prevent adiabatic heating and limited only by equipment tonnage limitations and excessive chilling. As known in the art, consolidation preferably yields a fully dense, fine-grain billet preferably having at least about 98% theoretical density. Prior to working the billet, a high temperature soak is typically performed in a manner that prevents excessive coarsening of the overall grain size that would excessively and undesirably increase flow stresses. In the practice of the present invention, a suitable soak has been achieved by simply preheating and holding the billet at its forging temperature for up to about five hours, though longer holding periods are also envisioned.

[0021] The billet is then hot worked (e.g., forged) to form a component having a desired geometry, followed by a supersolvus (solution) heat treatment. As taught by Yoon et al., under certain conditions an extended subsolvus annealing process or a low heating rate to the supersolvus heat treatment temperature may be desired to dissipate stored strain energy within the article and equilibrate the temperature of the component. Dissipation of stored strain energy can serve to reduce nonuniform nucleation tendencies of the superalloy, such that the tendency for critical grain growth in the component is also reduced. However, in the present invention such an extended subsolvus anneal step appears to be unnecessary. Instead, merely preheating the worked billet (forging) to within about 50°F to about 75°F (about 30°C to about 40°C) or so of the prior forging temperature is sufficient without any extended soak time.

[0022] The supersolvus heat treatment is then performed at a temperature above the gamma prime solvus temperature

(but below the incipient melting temperature) of the superalloy, to recrystallize the worked grain structure and dissolve (solution) the gamma prime precipitates in the superalloy. To accommodate furnace variability, a suitable supersolvus temperature is typically about 30°F to 70°F (about 15°C to 40°C) above the gamma prime solvus temperature of an alloy, though any temperature above the solvus temperature (but below the incipient melting temperature) is generally acceptable. Following the supersolvus heat treatment, the component is cooled at an appropriate rate to re-precipitate gamma prime within the gamma matrix or at grain boundaries, so as to achieve the particular mechanical properties desired. An example of a suitable cooling step includes controlled air cooling or controlled air cooling for a brief period followed by quenching in oil or another suitable medium. The component may also be aged using known techniques with a short stress relief cycle at a temperature above the aging temperature of the alloy if desirable to reduce residual stresses.

[0023] As noted above and well known in the art, in addition to grain recrystallization and solutioning gamma prime precipitates, heating the superalloy above its gamma prime solvus temperature causes grain growth (coarsening), typically resulting in grain sizes coarser than the original billet grain size, for example, coarser than about ASTM 9, such as in a range of about ASTM 2 to 9. To achieve mechanical properties desired for a gas turbine disk, uniform average grain sizes within a range of about two or three ASTM units are typically desired. Regions of the component with grain sizes in excess of about two to three ASTM units coarser than the desired grain size range are undesirable in that the presence of such grains can significantly reduce the low cycle fatigue resistance of the component and have a negative impact on other mechanical properties of the component, such as tensile and fatigue strength. For example, a component having a grain size range of about ASTM 5 to 8 is preferably free of isolated grains and small regions of grains coarser than ASTM 3 (though widely scattered grains slightly coarser may be tolerable), and free of significant regions coarser than about ASTM 6. As noted above, excessively large grains caused by critical grain growth can be avoided during working of the billet by maintaining strain rates below a critical (maximum) strain rate for the superalloy in accordance with Krueger et al. However, mechanical properties would be further promoted by improving the grain size distribution and achieving a finer average grain size, for example, in a range of about ASTM 7 to 9, more preferably about 8 to 10.

[0024] According to the present invention, improved grain size distribution and finer average grain size can be achieved by increasing the minimum strain rate of the strain rate window. Furthermore, the maximum strain rate can be increased to values previously associated with causing critical grain growth in a given superalloy, but without inducing critical grain growth, by increasing the carbon content of the superalloy above its conventional upper limit. In part, the effect of the increased carbon content is believed to be an increased pinning force that inhibits abnormal grain growth. Generally, finely dispersed carbides restrict grain boundary motion during supersolvus heat treatment, such that the grains are not permitted to grow excessively and/or randomly to the extent that critical grain growth occurs. From the investigations reported below, in addition to a more rapid forging process and improved properties, other benefits appear to be the ability to perform the forging operation at relatively low temperatures and under non-superplastic conditions ($m < 0.3$).

[0025] According to Krueger et al., the critical strain rate of a gamma prime nickel-base superalloy is composition, microstructure, and temperature dependent, and can be determined for a given superalloy by deforming test samples under various strain rate conditions, and then performing suitable supersolvus heat treatments. The critical strain rate is then defined as the strain rate that, if exceeded during deformation and working of a superalloy and accompanied by a sufficient amount of total strain, will result in critical grain growth after supersolvus heat treatment. According to the present invention, in which higher strain rates are identified as being capable of achieving a more controlled and finer average grain size after supersolvus heat treatment, strain rates below a minimum strain rate result in an average grain size that may be coarser than desired for optimal properties. As with the maximum strain rate identified by Krueger et al., the precise value for the minimum strain rate parameter of this invention appears to vary depending on the composition and microstructure of the superalloy in question. Strain rates for regions within large components can be predicted analytically by performing experiments on small laboratory specimens, and then using modeling techniques to predict local deformation behavior within the components.

[0026] The ability to improve grain size distribution and achieve finer average grain size by increasing the strain rate during forging is represented in Figure 1, which is a graph representing strain rate versus temperature and resulting average grain sizes observed with forged specimens of the conventional R88DT alloy ("Prior art forgings") and forged specimens of alloys based on the R88DT but whose compositions were modified to contain elevated carbon levels in accordance with this invention ("Fine grain forgings"). Specifically, the conventional and modified R88DT alloys differ in their carbon contents, with specimens of the conventional R88DT alloy containing about 0.045 to 0.060 weight percent carbon and specimens of the modified R88DT alloys containing greater than 0.060 weight percent carbon, preferably at least 0.065% to about 0.085% carbon, and possibly higher. The bars in Figure 1 represent a shift in acceptable ranges for strain rates and forging temperatures identified through investigations leading to the present invention. The upper extent of each bar represents the upper strain rate limit at which critical grain growth can be avoided in the specimens represented by that bar. From Figure 1, it can be appreciated that the modified R88DT specimens are able to be forged at much higher rates than the conventional R88DT specimens without critical grain growth (about 0.1 s^{-1} maximum versus about 0.010 s^{-1} maximum). Figure 1 also represents that significantly finer grains were obtained with the modified

R88DT specimens (about ASTM 7 to about ASTM 9) as compared to the conventional R88DT specimens (about ASTM 6 to about ASTM 8).

[0027] The parameters and effects represented in Figure 1 were determined through a series of investigations. In preliminary investigations, the relationship between final grain size and strain rate, including maximum (critical) strain rate, was evidenced from testing performed on subscale right circular cylinder (RCC) and double cone (DC) specimens. All specimens were formed from compositions based on the superalloy R88DT, which is disclosed in commonly-assigned U.S. Patent No. 4,957,567 to Krueger et al. as having a composition of, by weight, about 15.0-17.0% chromium, about 12.0-14.0% cobalt, about 3.5-4.5% molybdenum, about 3.5-4.5% tungsten, about 1.5-2.5% aluminum, about 3.2-4.2% titanium, about 0.5-1.0% niobium, about 0.010-0.060% carbon, about 0.010-0.060% zirconium, about 0.010-0.040% boron, about 0.0-0.3% hafnium, about 0.0-0.01 vanadium, and about 0.0-0.01 yttrium, the balance nickel and incidental impurities. The gamma prime solvus temperature of R88DT is estimated to be about 1950-2150°F (about 1065-1180°C), typically 2025-2050°F (about 1105-1120°C), for about 40 volume percent gamma prime. The actual chemistries of the specimens are summarized in the table below.

	"0.066% C Specimens"	"0.070% C Specimens"
Chromium	15.92 weight percent	15.86 weight percent
Cobalt	12.78	12.90
Molybdenum	3.92	3.93
Tungsten	3.98	3.90
Aluminum	2.13	2.16
Titanium	3.74	3.68
Niobium	0.70	0.67
Zirconium	0.40	0.039
Boron	0.014	0.014
Hafnium	0.00015	0.000039
Vanadium	0.0026	0.0026
Yttrium	< 0.0005	< 0.0005
Iron	0.04	0.03
Carbon	0.066	0.070
Nickel	Balance (+ impurities)	Balance (+ impurities)

[0028] From the above, it can be seen that the investigation evaluated two groups of experimental specimens alloyed to contain either about 0.066% or about 0.070% carbon.

[0029] Figures 2 and 3 plot, respectively, average ASTM grain size (ASTM Standard E 112) and ALA grain size (ASTM Standard E 930) versus strain rates for both groups of RCC specimens. All specimens were forged at a temperature of about 1850°F, 1875°F, or 1900°F (about 1010°C, about 1025°C, or about 1040°C), and at a strain rate within a range of about 0.00032 to about 1 sec⁻¹. Nominal strain levels were about 0.7%. Forging temperatures were selected on the basis of prior investigations with similar RCC and DC specimens of the conventional R88DT alloy (the basis for the conventional R88DT data represented in Figure 1). Though prior work reported by Huron, "Control of Grain Size via Forging Strain Rate Limits for R88DT," Superalloys 2000, September 17-21, 2000 (a publication of TMS), has shown that a strain rate of about 0.010 sec⁻¹ is an upper strain rate limit for specimens of the conventional R88DT alloy in order to avoid critical grain growth, critical grain growth was not observed in any of the experimental specimens at any of the tested strain rates, even at strain rates of about 1 sec⁻¹ (0.066% C specimens forged at 1875°F), which is a factor of one thousand over what was previously thought possible. In contrast, the data corresponding to the current lower strain rate practice of rates less than 0.010 sec⁻¹ evidence the entire grain size distributions, for both average and ALA, are shifted to coarser grain sizes, regardless of carbon content, attesting to the impact of the strain rate effect.

[0030] From the above results, it was concluded that the elevated carbon contents of the experimental specimens enabled forging at strain rates significantly higher than was previously possible with the conventional R88DT alloy, and even higher than thought possible for the high carbon alloys taught by Raymond et al., without encountering critical grain growth. It was further concluded that the higher strain rates resulted in a finer average grain size than was previously

possible with the conventional R88DT alloy. The average grain sizes achieved within the specimens reported in Figure 2 are sufficiently finer to have a beneficial and significant effect on low cycle fatigue life and other mechanical properties such as ultimate tensile strength (UTS), as well as processing considerations such as reduced noise during sonic inspections.

[0031] The data plot of Figure 3 evidences ALA grain size also shows improvement over current lower strain rate practice at rates less than 0.010 sec^{-1} . The data show that, even at higher carbon levels, the entire grain size distributions (both average and ALA) are shifted to coarser grain sizes, again attesting to the impact of the strain rate effect.

[0032] While the data plotted in Figures 2 and 3 are for specimens containing either 0.066 or 0.070 weight percent carbon, the trends evidenced in the graphs indicate that similar benefits and even further improvements are possible with higher carbon contents. Based on these results, a suitable range for the carbon content is believed to be about 0.065 to about 0.085 weight percent. Higher carbon contents are also believed to be possible, such as about 0.10 weight percent, with the upper limit being generally limited only by the potential detrimental impact of excessive carbon on other properties of the superalloy.

[0033] As noted above, with the present invention it is believed that higher strain rates can be employed to produce suitable forgings without the forging process being fully superplastic, in other words, forging can be performed with strain rate sensitivity values of less than about 0.3. This aspect of the invention is evident from Figure 4, which is a graph plotting the strain rate sensitivity value (m) and average grain size versus strain rate for the data of Figure 2 corresponding to the 0.066% C specimens forged at 1875EF. Figure 4 evidences that finer average grain sizes were achieved with strain rate sensitivity values of less than about 0.3, and therefore well below the minimum strain rate sensitivity taught by Yoon et al. and Raymond et al.

[0034] Figure 5 is a graph plotting average and ALA grain size versus forging temperature for the 0.070% C specimens. Figure 5 suggests that a trend also exists for finer and more uniform average grain sizes with decreasing forging temperature in the 0.070% C specimens. This trend was also found for the 0.066% C specimens, evidencing a broad processing window for high carbon contents and the potential benefits of lower forging temperatures.

[0035] In another investigation, high pressure turbine disks were forged and analyzed to further assess the above-described findings regarding the ability to obtain finer average grain size by increasing strain rates during forging. Three of the disks were formed from R88DT modified to have a carbon content of either about 0.066 or about 0.070 weight percent, and were produced by powder metallurgy, extrusion consolidation, forging, and supersolvus heat treatment at about 2080°F (about 1140°C). As understood by those skilled in the forging art, forging processes can be designed using simulation models to produce die shapes and achieve a forging press operation that controls the local strain and strain rate history of regions of a forging within desired parameters. Furthermore, because the forged disks of this investigation and the forged specimens of the previous investigations have an array of local strains and strain rates for a given macro overall strain rate based on the upset ratio of the workpiece, the forge rates for the disks and previous specimens can be compared on the basis of a maximum strain rate. Using this approach, the 0.066% C and 0.070% C disks evaluated in this investigation were forged using nominally isothermal processes designed to achieve maximum strain rates of about 0.032 sec^{-1} . The forging steps for the 0.066% C and 0.070% C disks were controlled on a local limit basis so that all regions of the forgings were at or below the 0.032 sec^{-1} upper limit.

[0036] The consistency with which finer grain sizes can be obtained with higher strain rates is evidenced in Figures 6 and 7, which are normal probability plots of the average and ALA grain size, respectively, of the three forgings (Forgings #2, #3, and #4) produced to have one of the above-noted 0.066% C, and 0.070% C compositions. Twenty measurements were obtained for each forging arrayed uniformly about the forging cross section. The data for the 0.066% C and 0.070% C forgings are plotted along with data obtained from a fourth disk of the same geometry, but formed from a conventional R88DT composition including a conventional carbon content of about 0.052 weight percent, and processed to the conventional strain rate limit of less than 0.010 sec^{-1} (Forging #1). Even with the complexity of geometric factors in a contoured forging, the improved practice of the invention demonstrates a finer mean average grain size by about 2 ASTM grain size numbers and an improvement in the mean ALA grain size by about 1 ASTM grain size number.

[0037] Based on these results, to achieve the benefit in complex contoured forgings where geometric factors drive local strain rate variations, it is believed that maximum strain rates of about 0.032 sec^{-1} and above, which correspond to strain rate sensitivity values of less than 0.3, should be employed to achieve the refined grain size throughout such forgings. As previously noted, it is very unlikely in a complex forging that a target maximum strain rate will be uniformly achieved in all areas of the forging, and variations in strain rate can be such that setting an absolute minimum strain rate in the forging is not practical. On the other hand, maximum strain rates capable of avoiding critical grain growth while achieving a finer grain size and distribution in accordance with the invention will inherently fall over a range. Therefore, a maximum strain rate can be set as a target within a range of suitable maximum strain rates for a given forging, in which an upper limit for the range is necessary to avoid critical grain growth and a lower limit of the range is necessary to avoid or minimize low strain areas that may not achieve sufficiently high strain rate work to obtain the desired fine grain size and distribution sought by the invention. Alternatively or in addition, the forging shape may be defined so that the high strain rate non-superplastically deformed regions are located in specific areas advantageous

to the part operation and life.

[0038] The average grain sizes achieved within the specimens reported in Figures 6 and 7 were concluded to be sufficiently finer to have a beneficial and significant effect on low cycle fatigue life and other mechanical properties such as ultimate tensile strength (UTS), as well as processing considerations such as reduced noise during sonic inspections. Such benefits are evident in Figures 8 through 10. Figure 8 is a bar graph evidencing the improved sonicability of the 0.066% C and 0.070% C forgings of Figures 6 and 7 as compared to the conventional 0.052% C forging of Figures 6 and 7. The data show a reduction of sonic noise levels of about 40%, indicating improved inspectability compared to the conventional current processing and chemistry.

[0039] Finally, Figures 9 and 10 are normal probability plots comparing the ultimate tensile strength and yield strength, respectively, of the 0.066% C and 0.070% C forgings of Figures 6 and 7 as compared to the conventional 0.052% C forging of Figures 6 and 7. Both measures of tensile capability (ultimate tensile strength and 0.2% yield strength) show a significant improvement of the mean value of about 9 to about 10 ksi (about 62 to about 69 MPa), when using methods and compositions of this invention as compared to currently existing methods and compositions.

[0040] In view of the above, the method of this invention makes possible the production of components from R88DT and similar gamma prime nickel-base superalloys that consistently exhibit a finer average grain size. While the benefits of the invention were described in reference to the R88DT superalloy processed from powder metal starting materials, other materials could be used including spraycast materials, cast and wrought materials, etc. Furthermore, gamma prime nickel-base superalloys having compositions sufficiently approximating that of R88DT to have similar mechanical properties as R88DT, such as low cycle fatigue life, are also believed to benefit from the processing and composition modifications of the present invention. An example of such an alloy is believed to be René 104 (U.S. Patent No. 6,521,175), with a nominal composition of, by weight, about 16.0-22.4% cobalt, about 6.6-14.3% chromium, about 2.6-4.8% aluminum, about 2.4-4.6% titanium, about 1.4-3.5% tantalum, about 0.9-3.0% niobium, about 1.9-4.0% tungsten, about 1.9-3.9% molybdenum, about 0.0-2.5% rhenium, about 0.02-0.10% carbon, about 0.02-0.10% boron, about 0.03-0.10% zirconium, the balance nickel and incidental impurities. Another notable example is NF3 (U.S. Patent No. 6,521,175), with a nominal composition of about 16.0-20.0 percent cobalt, about 8.5-12.5 percent chromium, about 1.5-3.5 percent tantalum, about 2.0-4.0 percent tungsten, about 1.9-3.9 percent molybdenum, about 0.04-0.06 percent zirconium, about 1.0-3.0 percent niobium, about 2.6-4.6 percent titanium, about 2.6-4.6 percent aluminum, about 0.02-0.04 percent carbon, about 0.02-0.04 percent boron, the balance nickel and incidental impurities.

[0041] While the invention has been described in terms of particular processing parameters and compositions, the scope of the invention is not so limited. Instead, modifications could be adopted by one skilled in the art, such as by substituting other gamma prime precipitation strengthened nickel-base superalloys with higher or lower gamma prime contents, or by modifying the preferred method by substituting other processing steps or including additional processing steps. Accordingly, the scope of the invention is to be limited only by the following claims.

Claims

1. A method of forming an article from a gamma prime precipitation-strengthened nickel-base superalloy having a gamma prime solvus temperature, the gamma prime precipitation-strengthened nickel-base superalloy containing greater than 0.060 weight percent carbon and being formed as a billet, the method being **characterized by**:

working the billet at a temperature below the gamma prime solvus temperature of the superalloy so as to form a worked article, wherein the billet is worked to undergo non-superplastic deformation and to achieve a maximum strain rate that is below an upper strain rate limit to avoid critical grain growth yet sufficiently high to control average grain size, wherein the upper strain rate limit is greater than 0.008 per second;
heat treating the worked article at a temperature above the gamma prime solvus temperature of the superalloy for a duration sufficient to uniformly coarsen the grains of the worked article; and
cooling the worked article at a rate sufficient to reprecipitate gamma prime within the worked article, wherein the worked article has an average grain size of not coarser than ASTM 7 and is substantially free of grains in excess of three ASTM units coarser than the average grain size.

2. The method according to claim 1, **characterized in that** the superalloy contains up to about 0.10% carbon.
3. The method according to claim 1 or claim 2, **characterized in that** the maximum strain rate is at least 0.010 per second.
4. The method according to claim 1 or claim 2, **characterized in that** the maximum strain rate is at least 0.032 per second.

EP 2 019 150 A1

5. The method according to any one of claims 1 through 4, **characterized in that** the upper strain rate limit is greater than 0.100 per second.
6. The method according to any one of claims 1 through 5, **characterized in that** the worked article has an average grain size in a range of about ASTM 7 to 10.
7. The method according to any one of claims 1 through 5, **characterized in that** the worked article has an average grain size of not coarser than ASTM 8.
8. The method according to any one of claims 1 through 5, **characterized in that** the worked article has an average grain size of about ASTM 8 to about ASTM 10.
9. The method according to any one of claims 1 through 8, **characterized in that** the superalloy contains, by weight, about 15.0-17.0% chromium, about 12.0-14.0% cobalt, about 3.5-4.5% molybdenum, about 3.5-4.5% tungsten, about 1.5-2.5% aluminum, about 3.2-4.2% titanium, about 0.5-1.0% niobium, at least 0.065-0.10% carbon, about 0.010-0.060% zirconium, about 0.010-0.040% boron, about 0.0-0.3% hafnium, about 0.0-0.01 vanadium, and about 0.0-0.01 yttrium, the balance essentially nickel and incidental impurities.
10. The worked article formed by the method of any one of claims 1 through 9, **characterized in that** the worked article is a turbine disk of a gas turbine engine, and after the cooling step the worked article has an average grain size of ASTM 8 to 10.

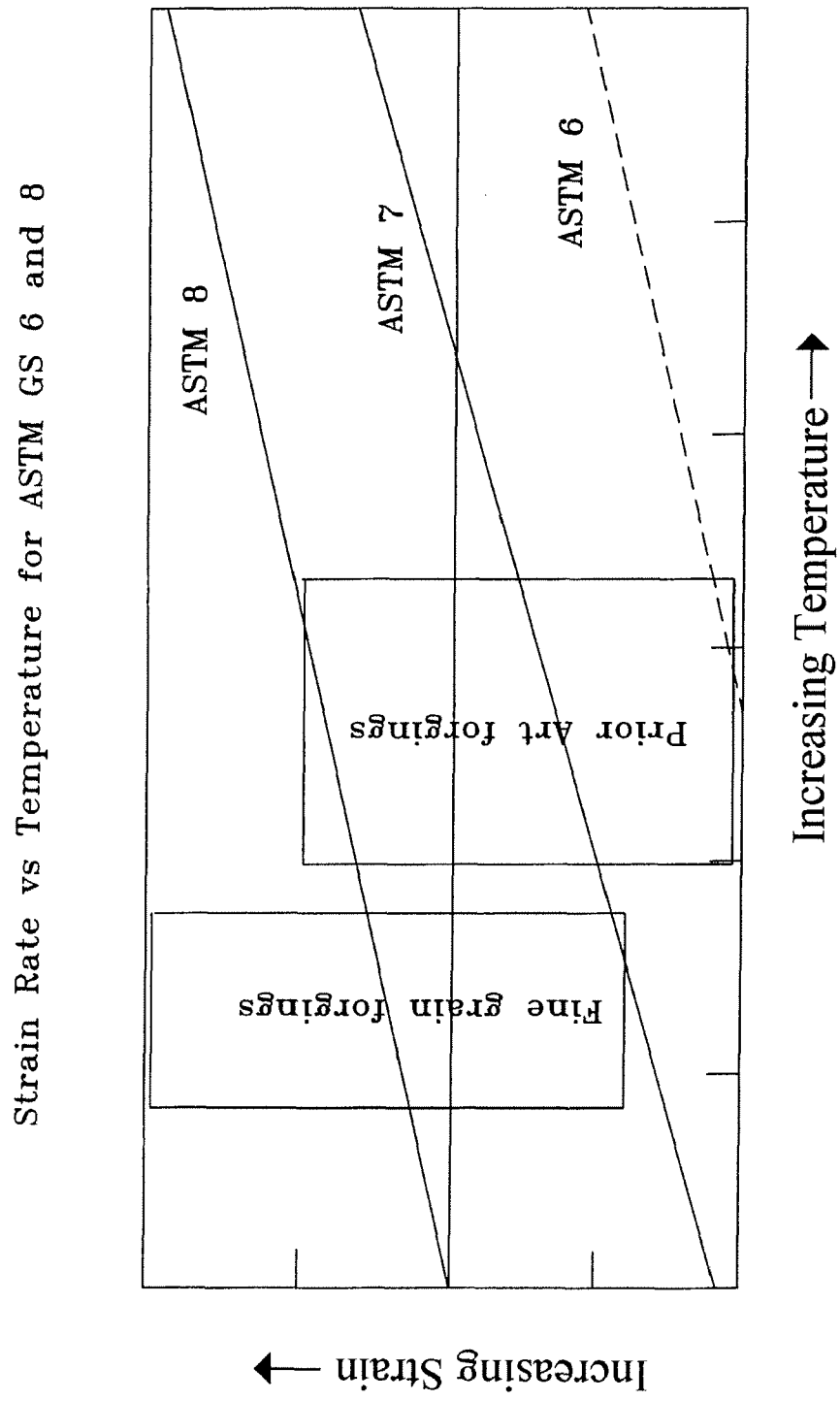


FIG.1

FIG.2
Normal Probability Plot

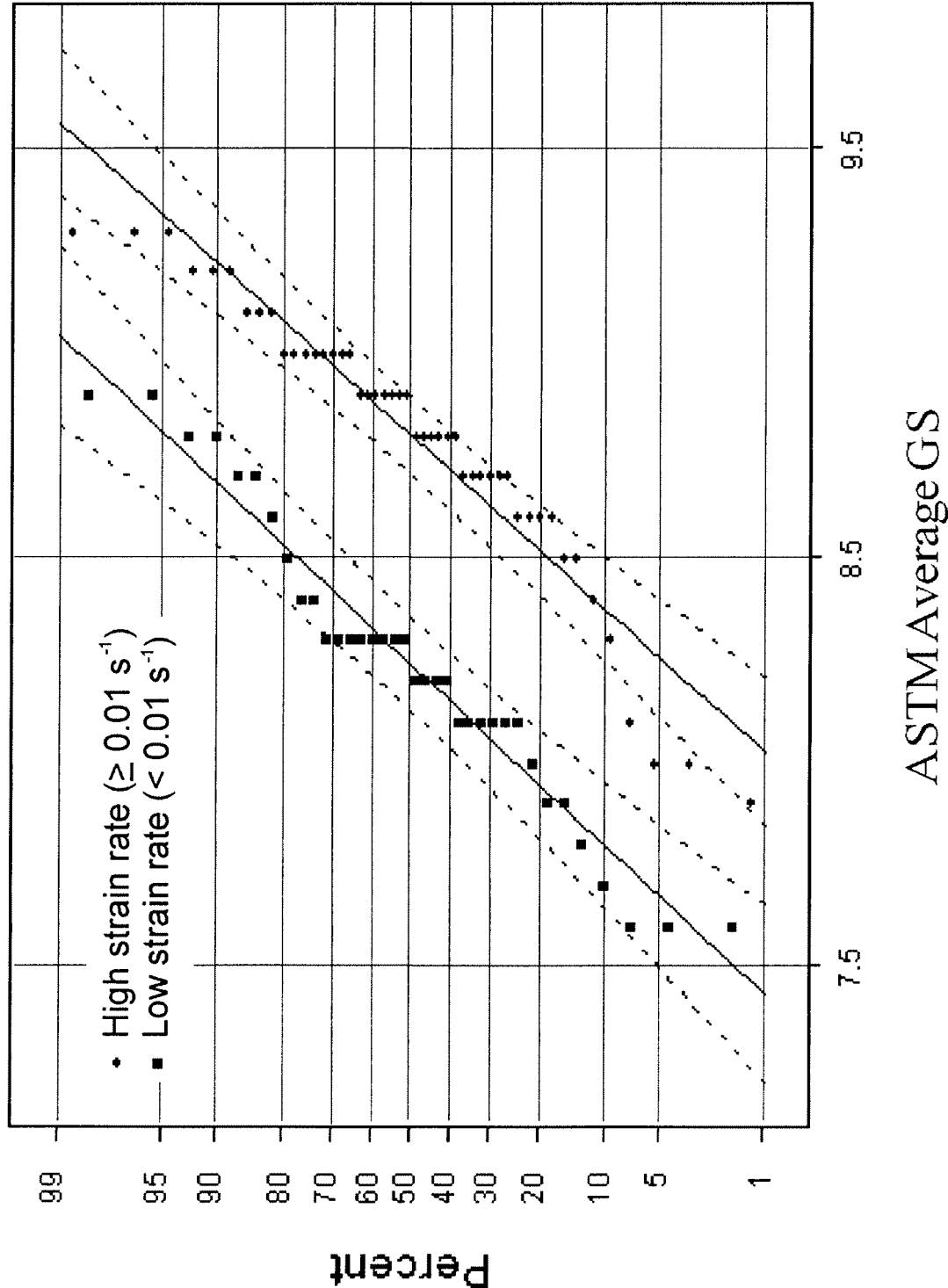


FIG.3

Normal Probability Plot

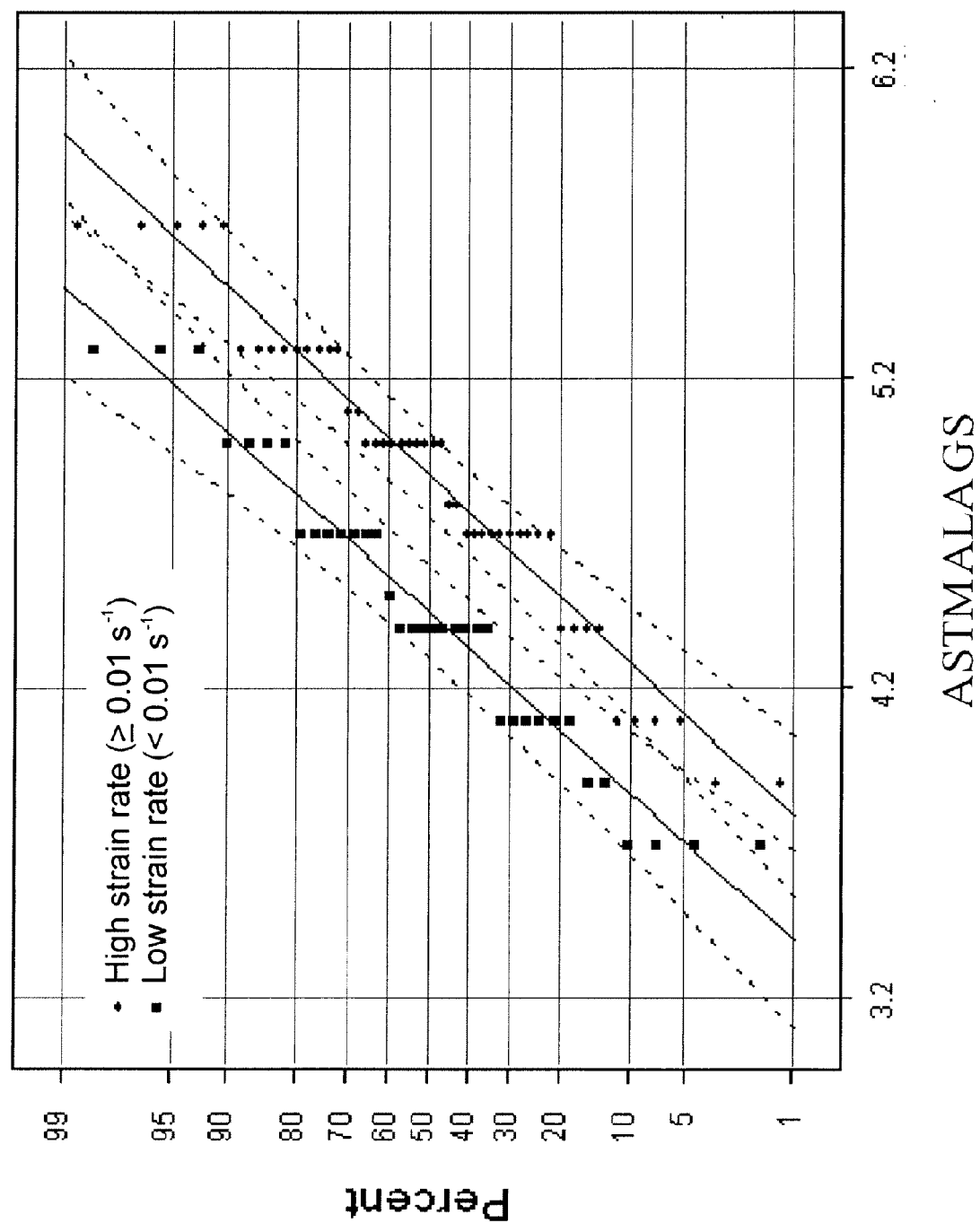
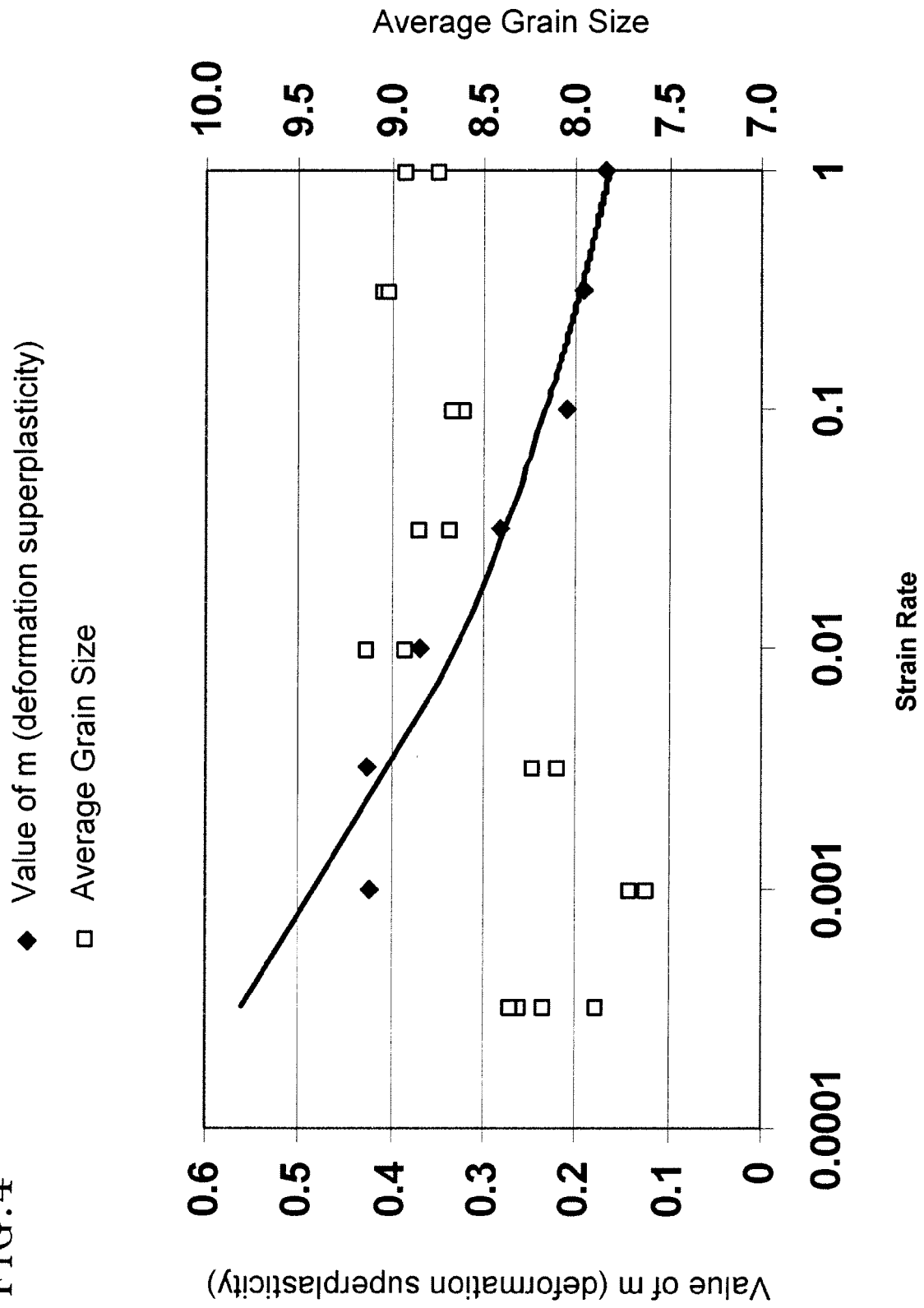


FIG. 4



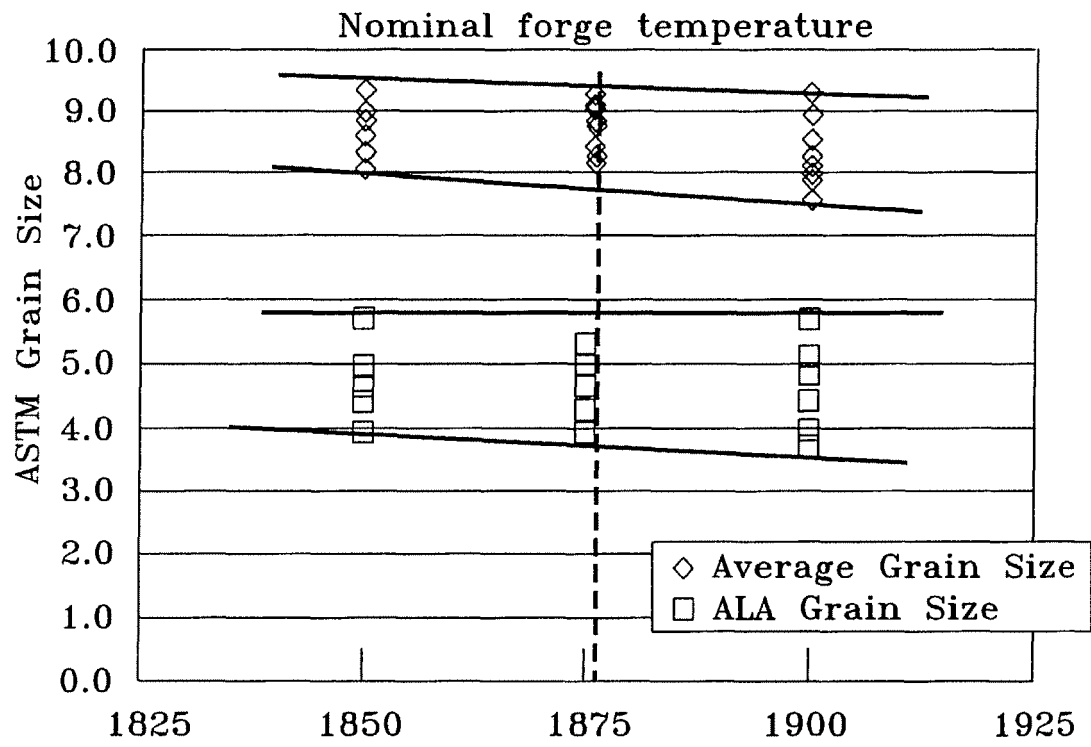


FIG.5

FIG.6

Forging	Strain Rate Limit	Carbon %
Forging #1	<0.010 sec-1	0.052
Forging #2	0.032 sec-1	0.066
Forging #3	0.032 sec-1	0.070
Forging #4	0.032 sec-1	0.070

Normal Probability Plot for avg By Forge rate

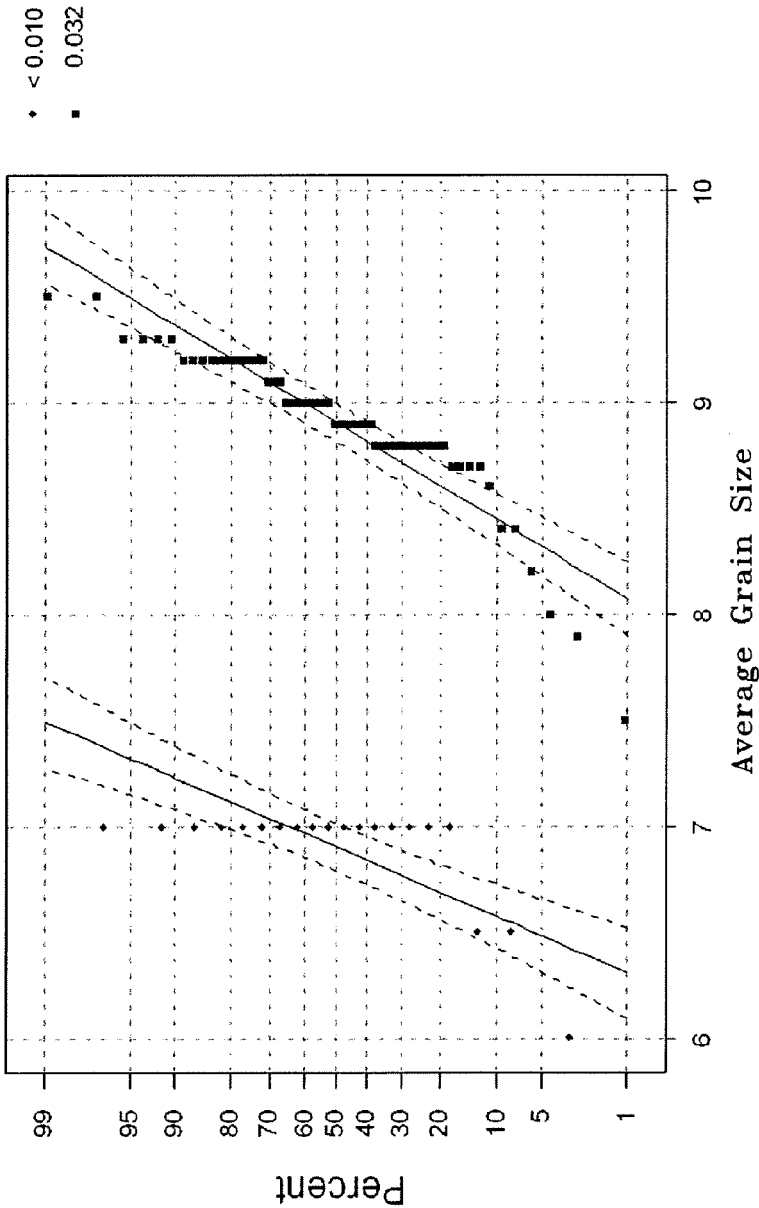


FIG. 7

Forging	StrainRate Limit	Carbon %
Forging #1	<0.010 sec-1	0.052
Forging #2	0.032 sec-1	0.066
Forging #3	0.032 sec-1	0.070
Forging #4	0.032 sec-1	0.070

Normal Probability Plot for ala By Forge rate

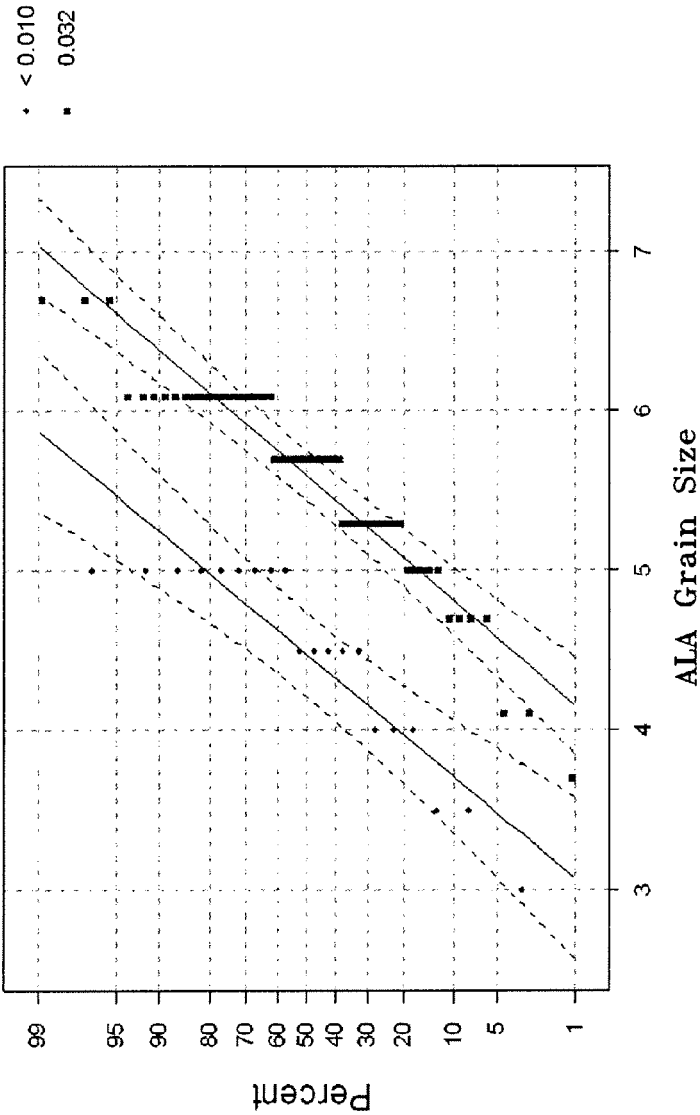


FIG. 8

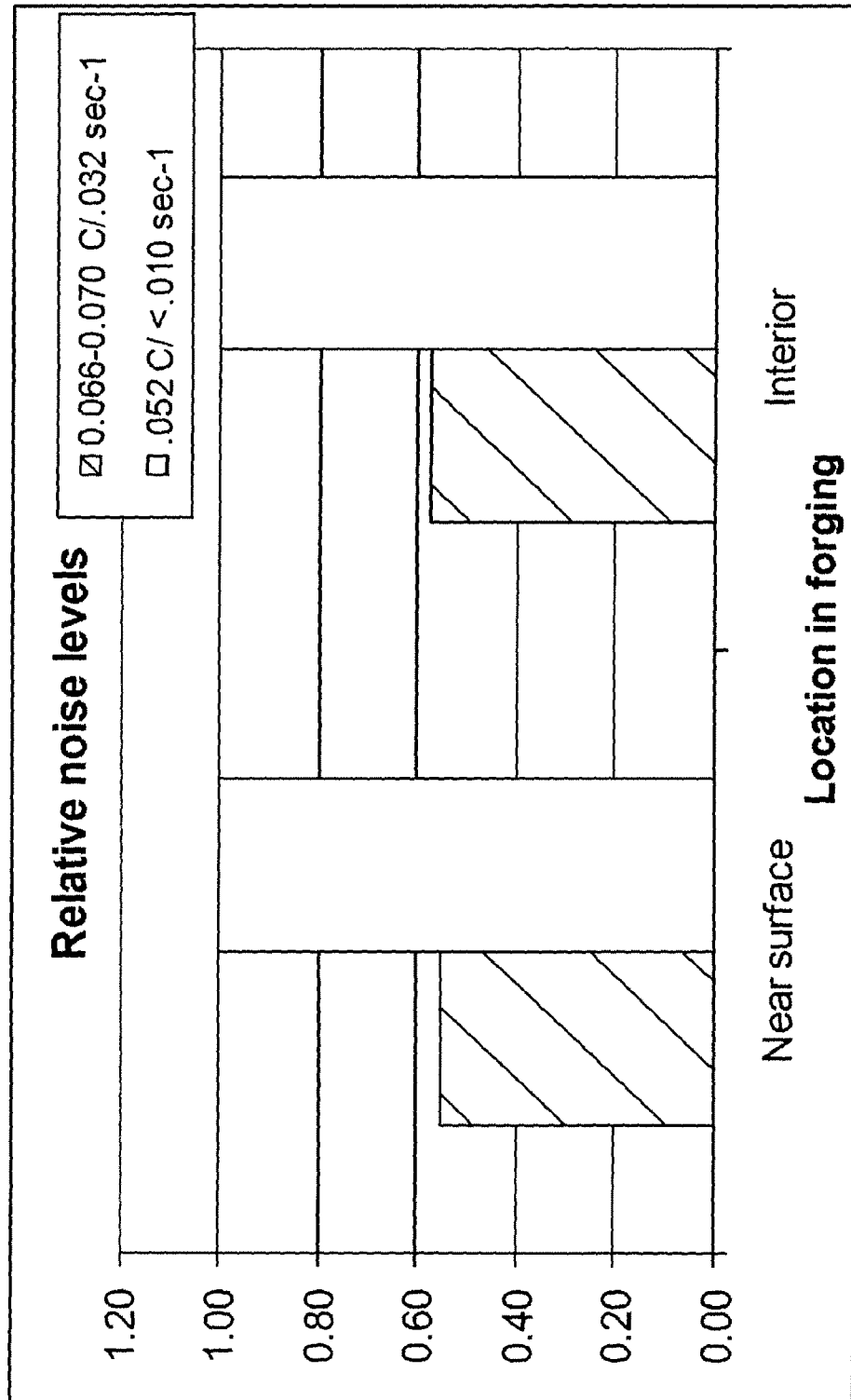


FIG. 9

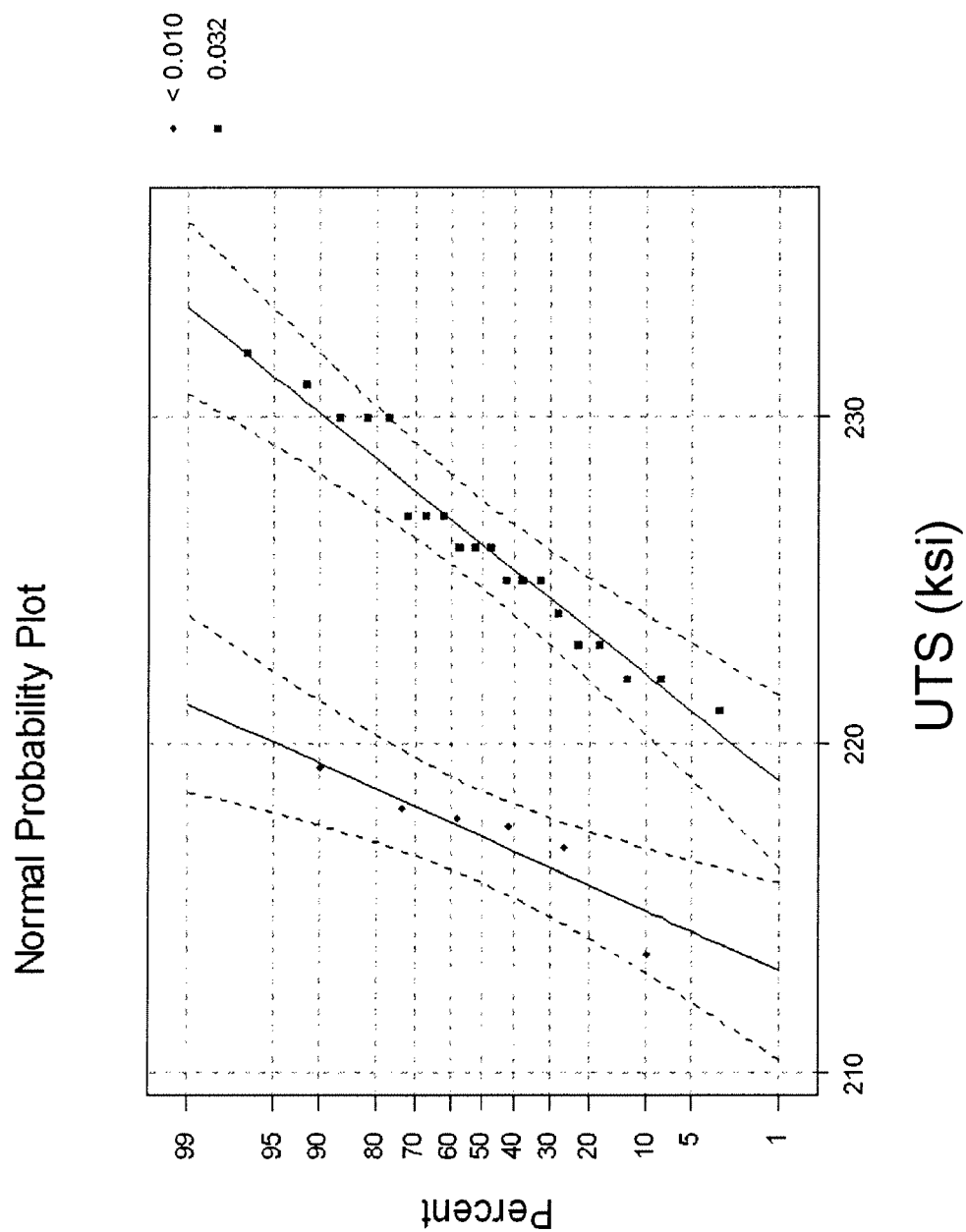
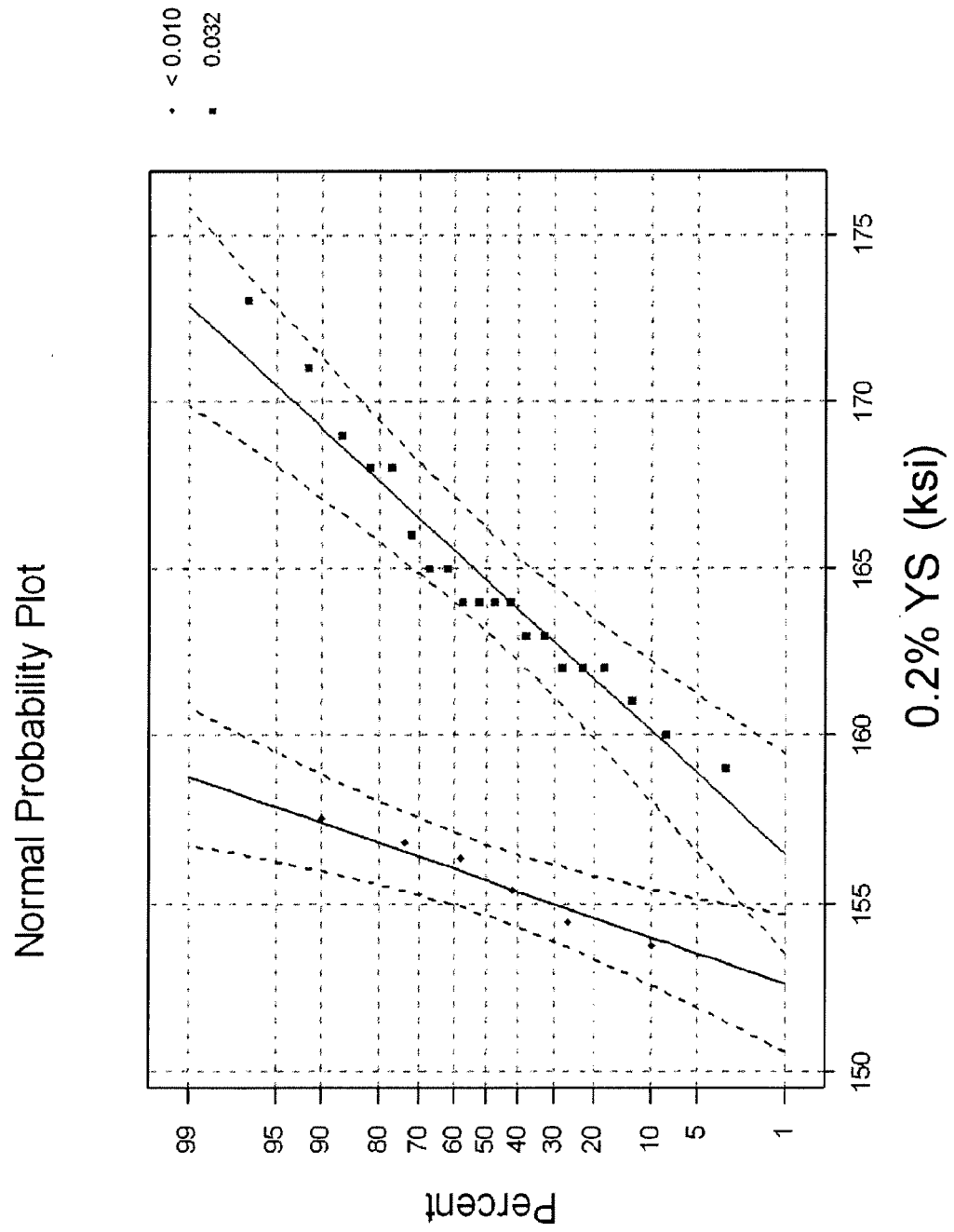


FIG. 10





European Patent
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EUROPEAN SEARCH REPORT

Application Number
EP 08 15 8579

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	US 6 059 904 A (BENZ MARK GILBERT [US] ET AL) 9 May 2000 (2000-05-09) * figure 4; table 1 * * column 1, line 53 * * column 2, lines 54-56,62-64 * * column 4, lines 49-55 * * column 6, lines 1-8,65-67 * * column 7, lines 10,11,21-23,52-55 * * column 8, lines 58-60 * -----	1-10	INV. C22C19/05 C22F1/10
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The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of the search 9 September 2008	Examiner Ugarte, Eva
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			

4
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09-09-2008

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