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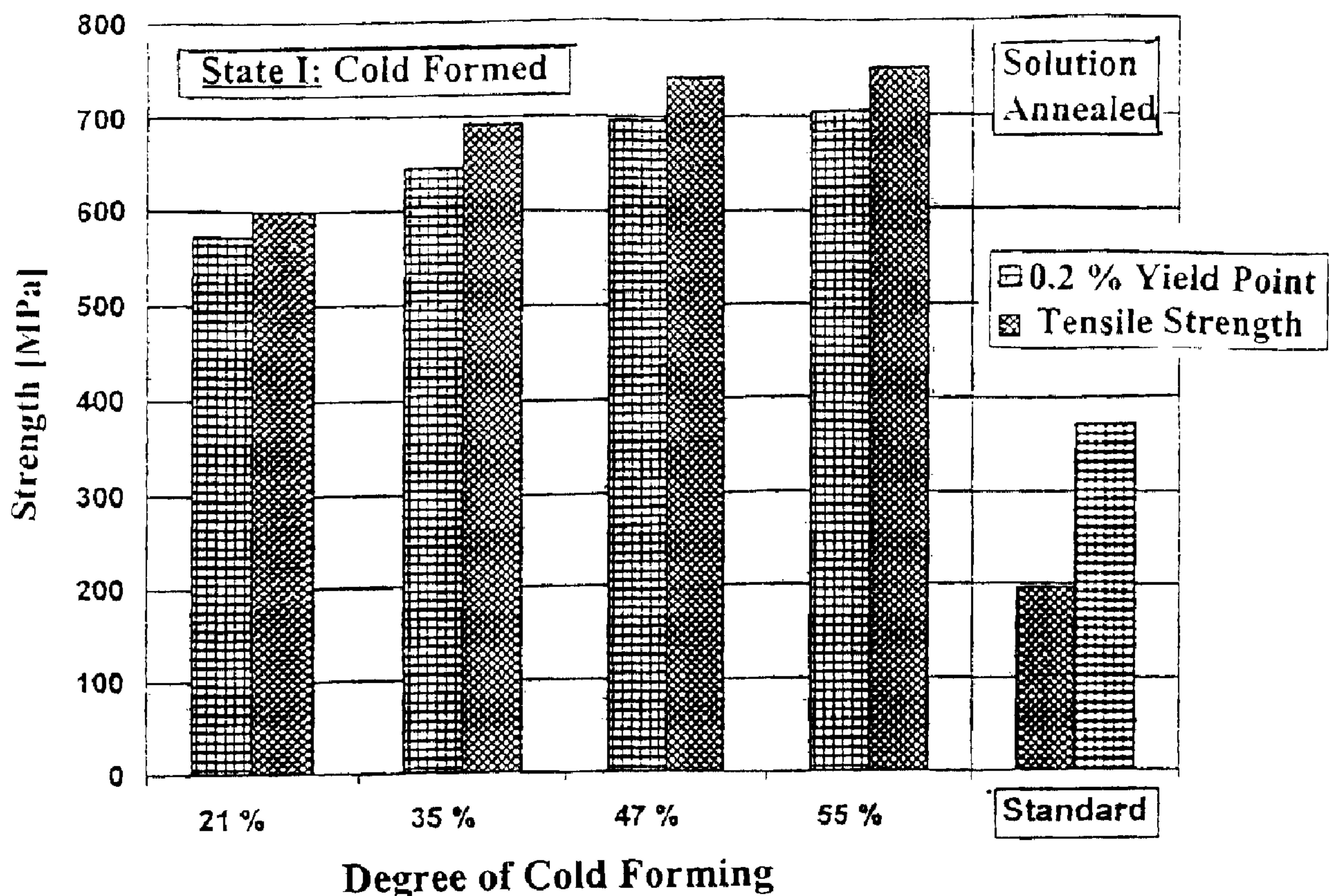
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(57) Abrégé/Abstract:

A material for the manufacture of parts and tools for use at elevated temperature, comprising an iron-based alloy comprising C, Si, Mn, Cr, Ni and N in certain concentrations and being cold formed to a hardness of at least 230 HB, a process for the manufacture

(57) **Abrégé(suite)/Abstract(continued):**

of the material and a hot working tool comprising the material. This abstract is neither intended to define the invention disclosed in this specification nor intended to limit the scope of the invention in any way.

ABSTRACT OF THE DISCLOSURE

A material for the manufacture of parts and tools for use at elevated temperature, comprising an iron-based alloy comprising C, Si, Mn, Cr, Ni and N in certain concentrations and being cold formed to a hardness of at least 230 HB, a process for the manufacture of the material and a hot working tool comprising the material. This abstract is neither intended to define the invention disclosed in this specification nor intended to limit the scope of the invention in any way.

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INERT MATERIAL WITH INCREASED HARDNESS FOR THERMALLY STRESSED PARTS

BACKGROUND OF THE INVENTION

1. Field of the Invention

[0002] The present invention relates to a material with high inertness, in particular high oxidation stability, and increased hardness for the manufacture of thermally resistant parts and tools.

2. Discussion of Background Information

[0003] According to DIN (German Industrial Standard) 50900, a reaction of a metallic material with its surroundings which causes a measurable change in the material, is defined as corrosion. Corrosion can occur with and without mechanical stress of the part, as well as after various kinds of chemical attack and at different temperatures.

[0004] A surface attack of objects is most often caused by an electrochemical corrosion in the presence of an ion conducting phase or by chemical corrosion and hot corrosion at elevated temperatures. A corrosion attack can also occur in molten media at elevated temperature, e.g., in liquid glasses, with a change in the surface of a metal part in contact therewith.

[0005] In modern technology, parts and tool parts are mostly exposed to a plurality of different stresses at the same time, of which in particular the thermal and mechanical stresses can act in an alternating or increasing manner. Accordingly, multiply intensified corrosion

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conditions exist which are possibly reinforced by a deformation of the zone close to the surface of the part.

[0006] Corrosion- and heat-resistant steels and alloys should have a cubic face-centered atomic lattice structure or an austenitic microstructure, respectively, also for a thermal resistance at temperatures above 600°C. In terms of alloy technology, this means that for increased strength and hardness at high temperatures, such materials have higher nickel and/or cobalt contents or are formed as nickel-based or cobalt-based alloys. However, for reasons of chemical corrosion they must have a chromium content of higher than 13 % by weight.

[0007] Although a material with a high nickel concentration invariably shows an increased mechanical strength and high material hardness, respectively, which improves the performance characteristics of parts and tool parts at high temperature, for economical reasons there is a desire to reduce the nickel content to below 36 % by weight and to increase the chromium content of the alloy to above 16 % by weight in order to increase corrosion resistance.

[0008] Although due to a high chromium concentration, an austenitic iron-based material having a nickel content of less than 36 % by weight can withstand, if necessary, in combination with further elements inhibiting corrosion, a corrosion attack at high temperatures, e.g., at 600°C and above, for a required minimum period, the material exhibits a low hardness and strength and a limited behavior under long period stressing. Despite these disadvantages, alloys, e.g., according to DIN material Nos. 1.2780 and 1.2782 and 1.2786, are used as tools for glass processing for reasons of cost effectiveness and for manufacturing reasons.

[0009] It would be desirable to have available a material of the type mentioned at the outset which has a hardness of higher than 230 HB and shows a high creep resistance and an improved fatigue strength behavior and corrosion resistance even at temperatures above 600°C.

[0010] It would also be desirable to provide a process for the economical production of

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a material for parts and tools which have improved performance characteristics as well as high hardness and increased corrosion resistance.

SUMMARY OF THE INVENTION

[0011] The present invention provides a material for the manufacture of parts and tools for use at elevated temperature. The material comprises an alloy having a composition of, in % by weight:

Carbon (C) 0.01 to 0.25

Silicon (Si) 0.35 to 2.5

Manganese (Mn) 0.4 to 4.3

Chromium (Cr) 16.0 to 28.0

Nickel (Ni) 15.0 to 36.0

Nitrogen (N) 0.01 to 0.29

provided that the nickel content of the alloy is equal to or higher than the value formed by the chromium content plus 1.5 silicon minus 0.12 manganese minus 18 nitrogen minus 30 carbon minus the numerical value of 6:

$$\text{Ni} \geq \text{Cr} + 1.5 \times \text{Si} - 0.12 \times \text{Mn} - 18 \times \text{N} - 30 \times \text{C} - 6$$

The balance of the alloy is iron (Fe) and accompanying elements and impurities. The material shows a hardness, provided by cold forming, of at least 230 HB.

[0012] It is noted that unless indicated otherwise, all weight percentages given in the present specification and the appended claims are based on the weight of the total composition. Moreover, all numerical values herein are approximate values.

[0013] According to one aspect of the material, the hardness of the material is higher than 250 HB, e.g., at least 300 HB.

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[0014] According to another aspect of the material, the nickel content of the alloy is higher by not more than 4.8 % than the value obtained according to the above equation.

[0015] In another aspect, the alloy comprises one or more (e.g., all) of the above elements in the following concentrations, in % by weight: C 0.02 to 0.20; Si 0.50 to 2.48; Mn 0.62 to 4.05; Cr 20.1 to 27.6; Ni 16.1 to 27.3; and N 0.014 to 0.23.

[0016] According to yet another aspect, the alloy comprises one or more (e.g., all) of the above elements in the following concentrations, in % by weight: C 0.04 to 0.15; Si 1.22 to 2.36; Mn 1.00 to 3.95; Cr 23.9 to 26.5; Ni 17.9 to 25.45; and N 0.018 to 0.20.

[0017] According to a still further aspect, the alloy comprises one or more (e.g., all) accompanying elements in the following concentrations, in % by weight: Molybdenum (Mo) less than 1.0; Vanadium (V) up to 0.5; Tungsten (W) up to 0.5; Copper (Cu) up to 0.5; Cobalt (Co) up to 6.5; Titanium (Ti) up to 0.5; Aluminum (Al) up to 1.5; Niobium (Nb) up to 0.5; Oxygen (O) up to 0.05; Phosphorus (P) up to 0.03; and Sulfur (S) up to 0.03. In this regard it is noted that the expressions “up to” and “less than” include the absence of the corresponding element; i.e., 0 % by weight.

[0018] The present invention also provides a process for producing a material for parts and tools for use at a temperature of up to 750°C. The process comprises the provision of an initial product from an alloy having a composition of, in % by weight, as indicated above and the subsequent cold forming of the initial product to a hardness of at least 230 HB.

[0019] In one aspect of the process, the initial product is formed by a process comprising hot forming and, subsequently, subjecting it to solution annealing or cooling down from the forming temperature, e.g., by forced cooling.

[0020] According to another aspect of the process, the cold forming is carried out over the whole circumference, and radially perpendicular to the longitudinal axis of the initial product.

[0021] According to yet another aspect, the degree of cold forming is such that the hardness of the material is higher than 250 HB, e.g., at least 300 HB.

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[0022] In a still further aspect of the process, the degree of cold forming is at least 6 %, for example, at least 12 %.

[0023] Also provided by the present invention is a hot working tool which comprises a cold formed material of an alloy having a composition of, in % by weight, as indicated above.

[0024] According to one aspect of the tool, the cold formed material has a hardness of at least 230 HB, e.g., higher than 250 HB.

[0025] In another aspect, the hot working tool of can be used at a working temperature of higher than 555 °C, e.g., at a working temperature of higher than 602 °C. For example, the working temperature may be up to 750°C.

[0026] The present invention additionally comprises a mold for machine pressed glass. The mold is made, at least in part, from the cold formed material as indicated above. Also provided is a tool in the glass industry which comprises this material, as well as a process for the manufacture of a part or tool for use at elevated temperature. The process comprises providing the above cold formed material and making it into said part or tool.

[0027] The advantages obtained according to the invention lie, in particular, in the synergy of corrosion chemical resistance of the selected alloy and the properties of the material that can be achieved with this chemical composition by means of cold forming. The cold forming or forming below the recrystallization temperature of the cubic face-centered austenite results in a strengthening of the material by a blocking of dislocations in the crystal lattice. An increase in hardness and an increase in the strength of the material according to the invention associated therewith is retained even at use temperatures of above 600°C, which is surprising to those of skill in the art. Recovery processes that would be expected to occur in the distorted lattice, such as, e.g., a thermally activated cross slip and a recombination of dislocations, cannot be observed within the usual time periods. In other words, contrary to the expectation of those skilled in the art, the hot strength that is increased by means of a cold forming of the material composed according to the invention is retained, even at high use temperatures of the part, because a high creep resistance of the steel

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improves the fatigue strength thereof. Particularly with increasing thermal stress, as is the case with a metal mold for the manufacture of utility glasses, strong temperature fluctuations and, thus, local changes in the volume of the material occur on the working surface thereof. It has been found that due to the hardness and hot strength of the material which are increased according to the invention, the local deformation or the deformation close to the surface, respectively, of the material, e.g., of a glass mold, occurs in the elastic area thereof, and that a fatigue crack formation, which occurs already with slight plastic deformations and can lead to the breakdown of the mold, is counteracted thereby.

[0028] In order to ensure an improved property profile of the material, it is important for it to remain in the stable austenitic region even during a cold forming, and to not exhibit any zones with strain-induced martensite. This is achieved according to the invention by the stated nickel and chromium concentration ranges and by the concentration range of nickel as a function of chromium, silicon, manganese, nitrogen and carbon. As has been shown, higher nickel contents may impair the fatigue strength behavior. However, with low nickel concentrations, there may be a sudden decrease in the austenite stability and the hot strength of the material. Essentially the same applies to the elements carbon and nitrogen, where in particular nitrogen increases the fatigue strength of the material.

[0029] The performance characteristics of parts and tools according to the invention can be improved if the material has contents of one or more alloying elements, in % by weight, of

C = 0.02 to 0.20, preferably 0.04 to 0.15

Si = 0.50 to 2.48, preferably 1.22 to 2.36

Mn = 0.62 to 4.05, preferably 1.00 to 3.95

Cr = 20.1 to 27.6, preferably 23.9 to 26.5

Ni = 16.1 to 27.3, preferably 17.9 to 25.45

N = 0.014 to 0.23, preferably 0.018 to 0.20.

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[0030] It should be noted here that, as is known *per se*, starting at a content of 0.52% by weight, cobalt can improve the hot strength of the material also in the alloy according to the invention.

[0031] Although the elements molybdenum, vanadium, tungsten, titanium and niobium increase the creep resistance of the material at high temperatures, and copper and aluminum represent classic hardening elements, in the material according to the invention these steel impurities should have a reliable concentration, because, as has been found, higher contents thereof reduce corrosion resistance, in particular during temporary contact with pasty glass, and impair glass transparency because of an occurring surface roughness of the mold. The reason for this has not yet been adequately established, but the acceptor atoms Na^+ , K^+ , Ca^{2+} , B^{3+} , Al^{3+} and Si^{4+} are among the hard Lewis acids, whereby a hot corrosion stress occurs in the mold after each glass forming.

[0032] Naturally, impurities can impair the material properties, so that for the accompanying elements and/or impurity elements the alloy according to the invention should show concentrations, in % by weight, of

Molybdenum (Mo)	less than 1.0
Vanadium (V)	up to 0.5
Tungsten (W)	up to 0.5
Copper (Cu)	up to 0.5
Cobalt (Co)	up to 6.5
Titanium (Ti)	up to 0.5
Aluminum (Al)	up to 1.5
Niobium (Ni)	up to 0.5
Oxygen (O)	a max. of 0.05
Phosphorus (P)	a max. of 0.03

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Sulfur (S) a max. of 0.03.

[0033] In the process according to the present invention for producing a material for parts and tools with high inertness, in particular high oxidation stability, and increased hardness under thermal stresses at a temperature of up to 750°C, an initial product is made from an alloy with a composition, in % by weight, of essentially

Carbon (C) 0.01 to 0.25

Silicon (Si) 0.35 to 2.5

Manganese (Mn) 0.4 to 4.3

Chromium (Cr) 16.0 to 28.0

Nickel (Ni) 15.0 to 36.0

Nitrogen (N) 0.01 to 0.29

provided that the nickel content of the alloy is equal to or higher, optionally by a maximum of 4.8% by weight, than the value formed by the chromium content plus 1.5 silicon minus 0.12 manganese minus 18 nitrogen minus 30 carbon minus the numerical value of 6:

$$\text{Ni} \geq \text{Cr} + 1.5 \times \text{Si} - 0.12 \times \text{Mn} - 18 \times \text{N} - 30 \times \text{C} - 6$$

the balance being iron (Fe) and accompanying elements and impurities. This initial product is subsequently further processed by cold forming to produce a material with a hardness of higher than 230 HB.

[0034] By means of a cold forming of the alloy according to the invention, the elasticity limit of the material can be increased to a tension level that is not reached even close to the working surface of the part or tool through a change in volume caused by alternating thermal stress. Accordingly, even in the area of the grain boundaries no zones occur that are plastically deformed during the temperature change, whereby a crack formation due to fatigue can be avoided. An attack at grain boundaries by chemical or hot corrosion can thus be largely avoided, so that, such as, e.g., with a glass mold, a high working surface or surface quality is retained over a long period even under high stress and with large production

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quantities. In comparison, conventional glass molds often show erosion of material at the grain boundaries of the microstructure after a short time in use, with erosions spaced in the range of a few μm . Thus, unevenness in the wave length range of visible light is imparted to the formed glass, which can result in reflection interferences and milky glass effects.

[0035] The corrosion resistance and hot strength can be further increased and a fatigue crack formation can effectively be suppressed if in the process according to the invention, a material having a hardness of higher than 250 HB, in particular 300 HB and higher, is formed by cold forming.

[0036] When an initial product with a composition according to the invention is formed by means of hot forming, is subjected to a solution annealing or cooled down, optionally in a forced manner, from the forming temperature, and cold formed, a material with a particularly homogeneous microstructure and improved corrosion resistance can be produced.

[0037] In particular for largely axially symmetrically shaped tools, such as bottleneck ingot molds and the like, it can be advantageous if the cold forming of the material is carried out over the whole circumference radially perpendicular to the longitudinal axis of the initial product.

[0038] For an increased quality of the product, the alloy of the invention advantageously contains one or more alloying elements in the following concentrations in % by weight

C = 0.02 to 0.20, preferably 0.04 to 0.15

Si = 0.05 to 2.48, preferably 1.22 to 2.36

Mn = 0.62 to 4.05, preferably 1.00 to 3.95

Cr = 20.1 to 27.6, preferably 23.9 to 26.5

Ni = 16.1 to 27.3, preferably 17.9 to 25.45

N = 0.014 to 0.23, preferably 0.018 to 0.2.

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[0039] Furthermore, the concentrations of the individual alloying elements, in % by weight, in the iron-based alloy may be as follows:

Carbon (C)	up to 0.25
Silicon (Si)	up to 2.5
Manganese (Mn)	up to 4.3
Chromium (Cr)	16.0 to 28.0
Nickel (Ni)	15.0 to 36.0
Nitrogen (N)	0.01 to 0.29

provided that the nickel content of the alloy is equal to or higher, optionally by a maximum of 4.8 % by weight, than the value formed by the chromium content plus 1.5 silicon minus 0.12 manganese minus 18 nitrogen minus 30 carbon minus the numerical value of 6:

$$\text{Ni} \geq \text{Cr} + 1.5 \times \text{Si} - 0.12 \times \text{Mn} - 18 \times \text{N} - 30 \times \text{C} - 6$$

the balance being iron (Fe) and accompanying elements and impurities. The alloy is strengthened to a material hardness of at least 230 HB, preferably higher than 250 HB, by cold forming the initial product made thereof, to result in a material for hot working tools with a working temperature of higher than 555°C, preferably higher than 602°C, in particular up to 750°C.

[0040] The use of the above-mentioned iron-based alloy as a tool material in the glass industry, in particular as a mold material for machine pressed glasses, is particularly advantageous with regard to product quality and cost-effective production.

[0041] The material according to the invention will be described in more detail on the basis of comparative test results.

BRIEF DESCRIPTION OF THE DRAWINGS

[0042] The present invention is further described in the detailed description which

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follows, in reference to the noted plurality of drawings by way of non-limiting examples of exemplary embodiments of the present invention, in which like reference numerals represent similar parts throughout the several views of the drawings, and wherein:

Fig. 1 shows the strength as a function of the degree of the cold forming of a material according to the invention at 604°C;

Fig. 2 shows the hardness curve at room temperature after a long-term thermal stress at 600°C.

DETAILED DESCRIPTION OF THE PRESENT INVENTION

[0043] The particulars shown herein are by way of example and for purposes of illustrative discussion of the embodiments of the present invention only and are presented in the cause of providing what is believed to be the most useful and readily understood description of the principles and conceptual aspects of the present invention. In this regard, no attempt is made to show structural details of the present invention in more detail than is necessary for the fundamental understanding of the present invention, the description taken with the drawings making apparent to those skilled in the art how the several forms of the present invention may be embodied in practice.

[0044] Fig. 1 shows the strength of the material according to the invention at a test temperature of 604 °C as a function of the extent of the cold forming. The test material was forged at a temperature of 1010 °C and cooled in a forced manner from the forming heat and subjected to a solution annealing at 1060 °C. A cold forming was carried out on parts of the material with a forming degree of 21 %, 35 %, 47 % and 55 %, respectively. Specimens for tensile tests were subsequently made from these materials. The strength tests, namely the determination of the 0.2 % yield point and the tensile strength, were conducted at a temperature of 604°C, keeping the specimens at this temperature for 20 minutes. For comparison, standard material was solution annealed at 1060 °C, and samples made therefrom were likewise tested at 604 °C. The bar chart in Fig. 1 clearly shows an increase in the strength values of the material as a function of the forming degree, wherein (not shown

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in the diagram) a large increase in strength is already present at a degree of cold forming of more than 6 %, in particular more than 12 %.

[0045] Fig. 2 shows the fatigue strength of the material according to the invention at a temperature of 600 °C, determined by a hardness test of the specimens in a cold state, compared with DIN materials Nos. 1.2083 and 1.4028.

[0046] The material according to the invention was smelted with a composition, in % by weight, of C = 0.08, Si = 1.7, Mn = 1.15, P = 0.01, S = 0.002, Cr = 24.8, Ni = 19.8, N = 0.02, Mo = 0.26, V = 0.09, W = 0.11, Cu = 0.12, Co = 0.4, Ti = 0.01, Al = 0.02, Nb = 0.001, O = 0.0029, and cast into a test block. This test block was hot formed to produce test material. A solution annealing was carried out on the test material at 1060 °C with a subsequent quenching in water. Thereafter specimens with the designation H 5 (unformed) and specimens with the designation H 525 (with a degree of cold forming of 35 %) were subjected to a long-term annealing at 600 °C. The comparison materials Nos. 1.2083 and 1.4028 were hardened in oil from 1020 °C, tempered at 630 °C and likewise subjected to the long-term annealing. After 45, 90, 140 and 180 hours, the test material was removed from the oven, allowed to cool, and the hardness of the material was determined. Thereafter, the samples were reinserted (with a temperature cycle stress). The comparison material H 5 showed an expected hardness behavior, whereas the material H 525 according to the invention, cold-formed at 35 %, exhibited an increased hardness of 315 HB and a high fatigue strength. At 600°C, no reduction of hardness and no creeping of the material could be detected even under alternating thermal stress. In contrast, a clear decrease in hardness was detected in the martensitic standard steels as a function of the annealing time of the samples.

[0047] It is noted that the foregoing examples have been provided merely for the purpose of explanation and are in no way to be construed as limiting of the present invention. While the present invention has been described with reference to an exemplary embodiment, it is understood that the words which have been used herein are words of description and illustration, rather than words of limitation. Changes may be made, within the purview of the appended claims, as presently stated and as amended, without departing from the scope

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and spirit of the present invention in its aspects. Although the present invention has been described herein with reference to particular means, materials and embodiments, the present invention is not intended to be limited to the particulars disclosed herein; rather, the present invention extends to all functionally equivalent structures, methods and uses, such as are within the scope of the appended claims.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A material of high inertness, high oxidation stability, and high hardness for components and tools to be loaded with a temperature of up to 750°C, the material consisting of an alloy having a composition in % by weight:

Carbon (C)	0.04 to 0.15
silicone (Si)	1.22 to 2.36
manganese (Mn)	1.0 to 3.95
chromium (Cr)	23.9 to 26.5
nickel (Ni)	17.9 to 25.45
nitrogen (N)	0.018 to 0.2

with the proviso that the nickel contents of the alloy is equal or higher than a value, which is formed from the contents of chromium plus 1.5 silicon minus 0.12 manganese minus 18 nitrogen minus 30 carbon minus the numerical value of 6;

$$\text{Ni} \geq \text{Cr} + 1.5 \times \text{Si} - 0.12 \times \text{Mn} - 18 \times \text{N} - 30 \times \text{C} - 6 ; \text{ and}$$

optionally one or more of the following elements present in concentrations in % by weight of:

molybdenum (Mo)	smaller than 1.0
vanadium (V)	up to 0.5
tungsten (W)	up to 0.5
copper (Cu)	up to 0.5
cobalt (Co)	up to 6.5
titanium (Ti)	up to 0.5
aluminium (Al)	up to 1.5
niobium (Nb)	up to 0.5

the remainder being iron (Fe) as well as impurities;

wherein the material exhibits a hardness of more than 230 HB formed by cold deformation of more than 6%.

2. The material according to claim 1, wherein the material exhibits a hardness of more than 250 HB.

3. The material according to claim 2, wherein the material exhibits a hardness of more than 300 HB.
4. The material according to any one of claims 1 to 3, wherein the contents of nickel of the alloy is greater by 4.8% by weight in maximum, than the value which is calculated in accordance with the equation as defined in claim 1.
5. The material according to any one of claims 1 to 4, which comprises concentration in % by weight of values for one or more impurity elements:

oxygen (O)	0.05 in maximum
phosphorus (P)	0.03 in maximum
sulphur (S)	0.03 in maximum.
6. A process for fabricating a material as defined in any one of claims 1 to 5, wherein the process comprises the step of:

forming a base product by cold deformation of more than 6% to provide a hardness of greater than 230 HB.
7. The process according to claim 6, wherein the degree of cold deformation is more than 12%.
8. The process according to claim 6 or 7, wherein the cold deformation is effected in radial perpendicular direction to a longitudinal axis of the base product.
9. The process of any one of claims 6 to 8, comprising the additional steps of:

hot forming and cooling of the base product before the cold deformation.
10. The process according to claim 9, wherein the cooling of the base product is effected by a solution treatment.
11. Use of an alloy on an iron basis for a hot-working tool in the glass industry, the alloy comprising the following elements in % by weight of:

Carbon (C)	0.01 to 0.25
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silicone (Si)	0.35 to 2.5
manganese (Mn)	0.4 to 4.3
chromium (Cr)	16.0 to 28.0
nickel (Ni)	15.0 to 36.0
nitrogen (N)	0.018 to 0.29

with the proviso that the nickel contents of the alloy is equal, or higher by 4.8% in maximum, than a value, which is formed from the contents of chromium plus 1.5 silicon minus 0.12 manganese minus 18 nitrogen minus 30 carbon minus the numerical value of 6; $Ni \geq Cr + 1.5 \times Si - 0.12 \times Mn - 18 \times N - 30 \times C - 6$; and

optionally one or more of the following elements present in concentrations in % by weight of:

molybdenum (Mo)	smaller than 1.0
vanadium (V)	up to 0.5
tungsten (W)	up to 0.5
copper (Cu)	up to 0.5
cobalt (Co)	up to 6.5
titanium (Ti)	up to 0.5
aluminium (Al)	up to 1.5
niobium (Nb)	up to 0.5

the remainder being iron (Fe) as well as impurities;

wherein the alloy has a material hardness of more than 230 HB by cold deformation of more than 6%.

12. The use according to claim 11, wherein the alloy has a material hardness of more than 250 HB.

13. The use according to claim 11 or 12, wherein the degree of cold deformation is more than 12%.

14. The use according to any one of claims 11 to 13, wherein the material for the hot-working tool is a die material for machine pressed glass with a working temperature of higher than 558°C.

15. The use according to claim 14, wherein the working temperature is higher than 602°C.

16. The use according to claim 15, wherein the working temperature is higher than 750°C.

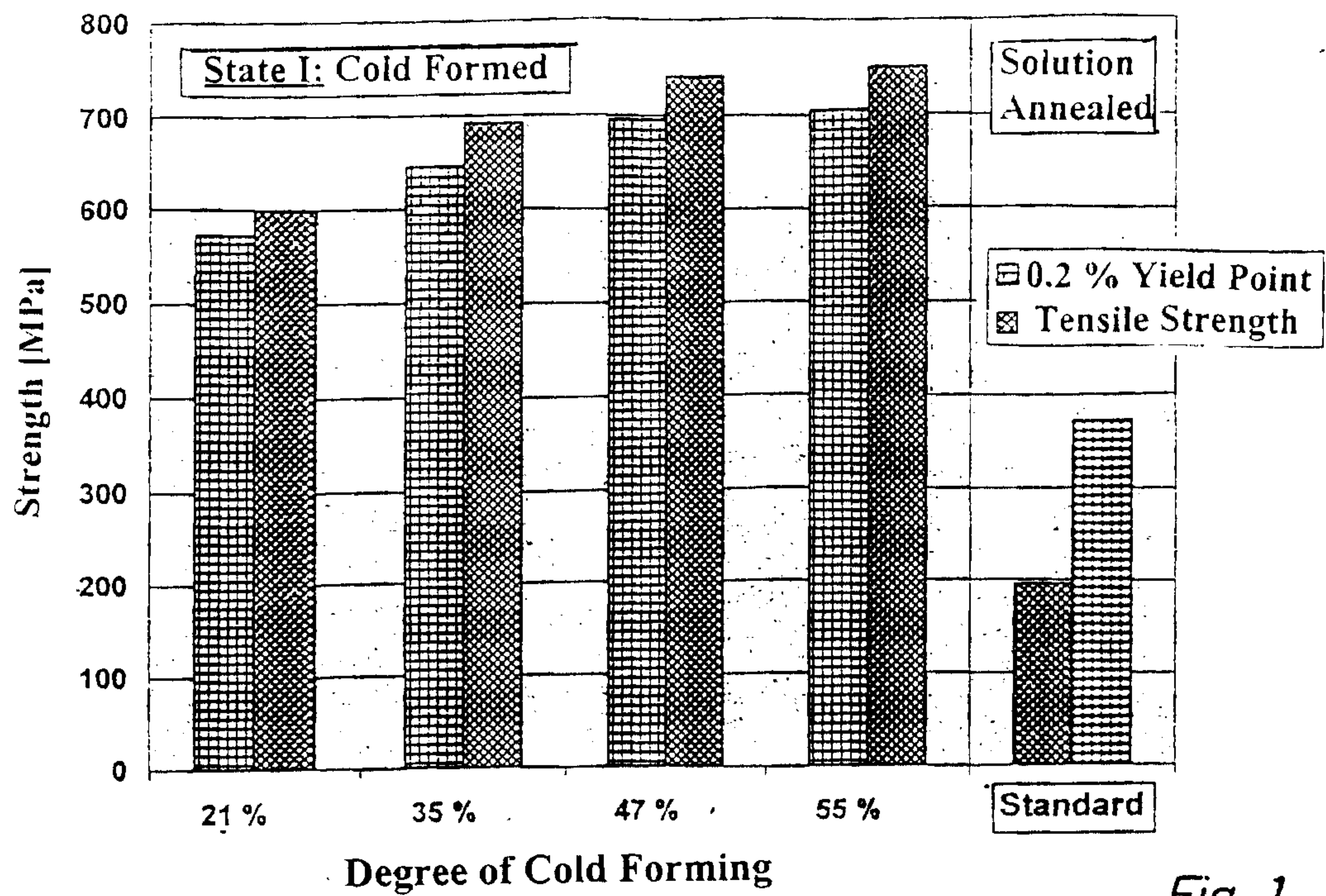


Fig. 1

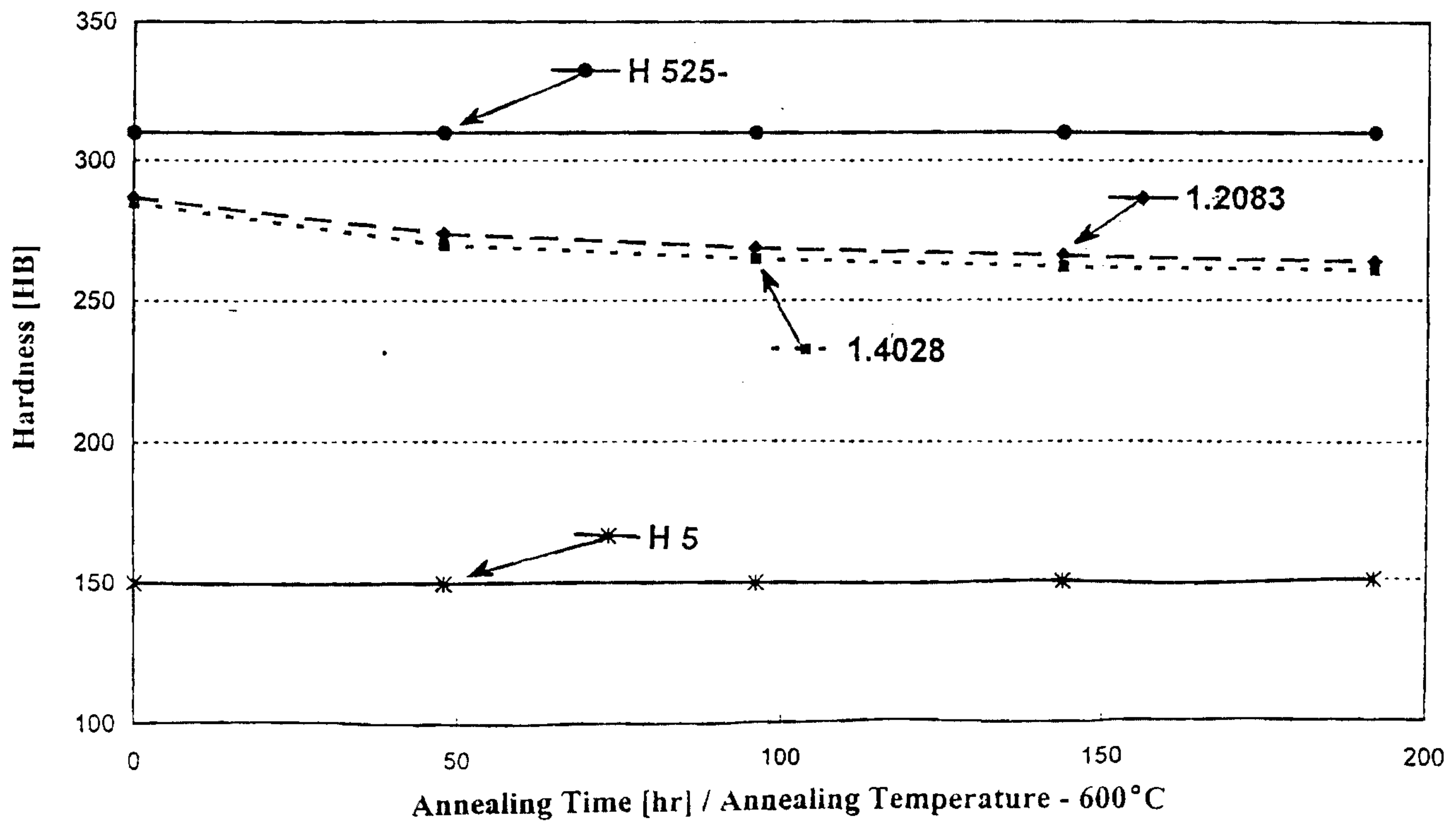


Fig. 2

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