(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 3 054 026 A1

(12) # EUROPEAN PATENT APPLICATION

(43) Date of publication:
10.08.2016 Bulletin 2016/32

(21) Application number: **15153854.3**

(22) Date of filing: **04.02.2015**

(51) Int Cl.:
C23C 8/02 (2006.01)
C21D 1/09 (2006.01)
C21D 6/00 (2006.01)
C23C 8/38 (2006.01)
C23F 17/00 (2006.01)

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR
Designated Extension States:
BA ME

(71) Applicant: **VitKovice Cylinders a.s.**
70600 Ostrava-Vitkovice (CZ)

(72) Inventor: **Kucera, Pavel**
73911 Frydlant nad Ostravici (CZ)

(74) Representative: **Malusek, Jiri**
Mendlovo namesti 1 a
603 00 Brno (CZ)

(54) **Method of combined surface treatment of tool steels**

(57) Method of combined surface treatment of tool steels where the functional portions of a tool undergo laser quenching followed by plasma nitriding in the subsequent step.

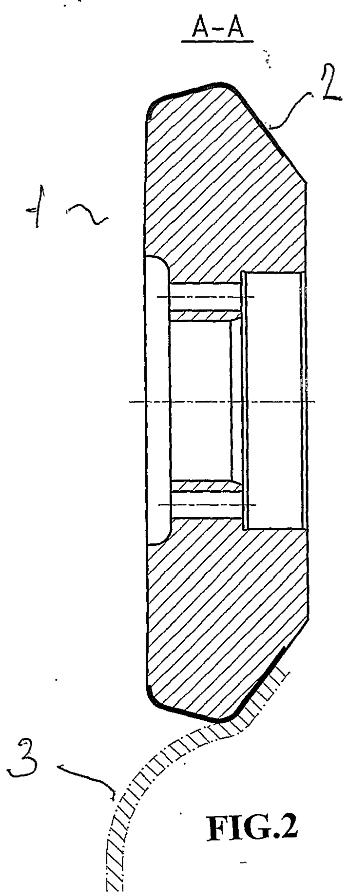

FIG.2

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Description

Field of the invention

[0001] The invention relates to a method of combined surface treatment of tool steels, particularly those used in the manufacture of forming tools.

Background of the invention

[0002] Tool steels have assumed an irreplaceable role in the engineering industry. Diverse surface treatment methods can be used with the aim to influence both the performance and the life properties of various tools. This particularly applies to forming tools where it is possible to considerably enhance the above properties by means of a suitable surface treatment. In the past, the surface properties of the forming tools used to be enhanced by the application of carbidic surface layers or by the application of deposited coatings based on TiN and/or TiCN.

[0003] There is a plurality of more up-to-date methods, one of them being the so called **CVD** (Chemical Vapour Deposition). Typically, the chemical vapour deposition is carried out under high temperatures (1,000 - 1,200°C). The advantages achieved by this method include the formation of deposited coatings having high density and thermal stability and featuring an excellent adhesion between the base material and the coating itself, the capability of depositing coatings onto workpieces having complex shapes and the low acquisition and operating costs. The drawbacks of this method include the adverse influence on the structure of base materials (resulting in the degradation of mechanical properties of the same) due to the high process temperature levels, the existence of residual stresses inside the coating layer (due to different thermal expansivity coefficients), high energy and time demands, adverse environmental effects caused by the process gases used and undesirable rounding of sharp edges.

[0004] Another method, which is known as **PVD** (Physical Vapour Deposition), is characterized by relatively low process temperatures below 500°C. Herein, the deposited coating layers are formed under reduced pressure (0.1-1.0 Pa). This method is preferably used for depositing coating layers onto sharp edges (having a fillet radius smaller than 20 μm). The typical properties of such coating layers include high durability and low friction coefficients. Besides that, combinations of different coating materials within a single layer can be used and precise thickness settings can be selected. The drawbacks of this methods include a relatively complex vacuum system and the so called shadow effect. Moreover, a coating having an uneven thickness distribution can form on the surfaces that do not lie in the direction of the particles.

[0005] Another up-to-date heat treatment method used for enhancing the surface properties of materials is **laser quenching**. A laser beam impinges on a surface layer consisting of the material to be quenched. This causes the surface layer to be rapidly heated up in order to reach a certain temperature, typically closely below the respective melting point (900 - 1,400°C). As such temperature is reached, the material structure undergoes austenitic transition. The incident point of the laser beam is being continuously shifted in the feeding direction, thus causing the heated spots to be rapidly cooled down through the heat transfer into the surrounding material. This enables a very fine carbidic structure to form which include short martensite needles and having small grain size. Such structure will cause the hardness of the surface layer to increase without compromising the core toughness and initiating any creation of fissure cracks. The main advantage of the laser quenching method consists in that it enables only those locations to be hardened where an increased durability is required without affecting the original material properties in the remaining portions or sections of the respective workpiece. Besides that, an environmentally friendly process is concerned.

[0006] Surface treatment methods based on the laser quenching technology are described in multiple patent documents. The applicability of the laser quenching method is described, for example, in the documents EP 0130749 B1 or GB 202838. Moreover, the document WO96/28574 describes the use of the laser quenching method for treating complex workpieces, in the present case for treating the inside corners of the workpieces. The drawbacks of the last-mentioned method consists in the related high acquisition and operating costs and in the existence of the problems connected with the treatment of highly reflexive materials.

[0007] At the present time, the **plasma nitriding** technology is considered to be one of the most progressive surface treatment technologies used in the engineering industry. A nitriding atmosphere consists of a mixture of nitrogen and hydrogen and the nitriding process is typically carried out in the temperature range from 500 to 550°C. The quality of the nitrided surface layer depends not only on the process temperature but also on the chemical composition of the steel being treated, on the quality of the surface finish of the respective tool and on the physical parameters of the nitriding process, such as the voltage, duration, pulse length and pressure of the gas mixture. This chemical heat treatment is used for increasing the surface hardness, corrosion resistance and fatigue strength. Further advantages include high levels of process accuracy and stability, reduced consumption of gas as well as the possibility of nitriding tool having final dimensions without necessitating the same to be subsequently machine finished (by grinding or the like). A substantial disadvantage, however, consists in the depth limitation of the nitrided surface layers, which corresponds to 0.3 mm of the base material, and in the related wear rate of such rough nitrided surface layer. The nitrided surface layer remains sufficiently stabile under the temperatures up to about 600 °C. After exceeding this temperature limit, the properties of such surface layer, particularly the abrasion resistance thereof, gradually degrade.

[0008] The plasma nitriding method is mentioned, for example, in the documents JP2013-234370 or KR100661130 B1. The document US 5536549 B1 discloses the application of this method for treating a special surface of a magnetic recording medium wherein the base metal of the respective disc-type carrier is an austenitic steel.

[0009] The object of the invention is to present a method which would enable an extended service life of the functional surfaces of the forming tools made of tool steels to be achieved. Such method should be easily applicable and, besides that, it should enable the desired structure and properties of the steel layer under the treated surface to be preserved in order to ensure the repeatability of the method after removing a worn out surface layer.

Disclosure of the invention

[0010] The above drawbacks are largely eliminated by the proposed method of combined surface treatment of tool steels according to the present invention, wherein the functional portions of a tool undergo laser quenching followed by plasma nitriding in the subsequent step. The above mentioned steps can be applied in the given order and with an arbitrary time interval.

Overview of the figures

[0011] The present invention will be further explained by means of the accompanying drawings wherein Fig. 1 is a front view showing a spin forming tool used for shoulder and neck forming in the manufacture of blanks for steel cylinders, Fig. 2 is a sectional view showing the spin forming tool from Fig. 1 and Fig. 3 is a diagram representing the difference between the service lives of multiple spin forming tools before and after undergoing a combined surface treatment based on the method according to the invention, respectively.

Preferred embodiment of the invention

[0012] The tool 1 shown in Figs. 1 and 2, namely the spin forming tool, is first machined from a forged piece in order to obtain its final shape and subsequently heat treated in order to obtain the desired hardness within its whole volume. Since the whole-volume heat treatment based on the hardness range according to the respective shop drawing would not be, as such, sufficient for the subsequent use in a hot forming process, such as spin forming one, with respect to the desired abrasion resistance, it is necessary to additionally enhance the surface resistance properties of the blank.

[0013] In the given case, such objective can be achieved by means of the method of combined surface treatment according to the present invention, namely in that first the functional surface 2 is heat treated using the laser quenching technology. Shape of the treated blank 3 is Ilustrated for information. During this treatment process, the structure of the surface layer as well as that of a thin subsurface layer is transformed into a very fine martensitic structure exhibiting very high hardness values up to 55 HRC. In this way, an overall thickness of the through-hardened layer of up to 1.3 mm can be obtained. The laser quenching process is implemented by means of high-power filament focused laser devices.

[0014] The above described surface heat treatment step is followed by the chemical heat treatment one consisting in the application of the plasma nitriding technology. During this subsequent processing step, very fine and hard nitrides form in the surface layer of the steel blank due to the fact that the layer is saturated with nitrogen. This causes an additional thin surface layer to form. The latter layer has at most 0.35 mm in thickness, the hardness of the same reaching up to 62 HRC. This process is carried out in the temperature range of 520 - 540°C for 24 hours. The respective process atmosphere contains a gas mixture consisting of H_2 and N_2 in the ratio of 80:20. The surface area, which is highlighted in bold and provided with the reference numeral 2, is exactly that area, which undergoes the treatment and through which the tool is brought into contact with the forged piece, i.e. with the blank of the steel cylinder to be closed in its top portion by means of the process of shoulder and neck forming. During processing, the forged piece is driven in rotation, thus imparting rotational movement to the forming tool that constitutes a driven, freely rotating unit.

[0015] The surface treatment method according to the invention was applied in the manufacture of a forming tool which is referred to as "spin forming tool". Such a type of forming tool is used for the so called spin forming which is a method used for shoulder and neck forming in the manufacture of backwardly extruded high-pressure steel cylinders (by way of illustration, the method, which is used for all the types of steel cylinders, may be compared with the shoulder and neck forming process used in the manufacture of necked tubes, the latter having one end provided with a necked spherical cap).

[0016] The forming tool is made of the hot work tool steel QRO 90 Supreme, the most similar equivalent of the latter being the steel X32CrMoV33. Hence, the typical base material used for manufacturing the tool blank is the above mentioned steel QRO 90 Supreme. Before being employed in the manufacturing process, the spin forming tool is preheated to reach a temperature ranging between 180 and 220°C. Such elevated temperature ensures a better adhesion during the initial phase of the forming cycle. Afterwards, the working cycle in itself commences. This main cycle consists of heating up the blank to be formed to reach a temperature ranging between 1,000 and 1,200°C, bringing the hot blank, which is continuously rotating around its centre line, into contact with the spin forming tool and in subsequent pressing the spin forming tool against the blank, thus causing the material of the latter to "flow" in the direction defined by the swinging movement of the spin forming tool. During the forming process, the material being formed is continuously reheated by means of burners in order to be held at the above mentioned forming temperature. After the completion of the forming process, the surface of the spin forming tool is cleaned by means of a high-pressure jet.

[0017] Until recently, solely the plasma nitriding method has been applied as a surface treatment technology with the aim to improve the characteristics influencing tool life. This technology by itself, however, was not able to ensure any improvement of the total tool life. Instead, it only enabled a temporary improvement to be achieved. This means that the thickness of the nitrided layer containing highly abrasion resistant nitrides was used up after having shaped 1,800 - 2,000 workpieces, on an average, and afterwards solely the base material of the tool was used for shaping the workpieces which led to the occurrence of local deformations and increased abrasive wear of the base material of the spin forming tool as well as to the development of a "sticking" effect between the latter material and that of the workpiece. By means of the laser quenching step, which has been incorporated into the technological process so as to precede the plasma nitriding one, a very fine martensitic structure can be obtained. The latter structure enables an additional abrasion resistant sublayer to form that remains available even after a complete depletion of the nitride layer due to abrasive wear. Simultaneously, the martensitic transformation taking place during the laser quenching process, ensures a significant grain refinement within the hardened layer which is achievable owing to the typical rapid chilling effect in the course of the process. This grain refinement is also a significant prerequisite for creating more favourable conditions for the subsequent nitriding step because it enables a stronger diffusion of nitrogen into the material to be achieved due to the smaller size and increased number of the grains.

[0018] Once the contact surface 2 of the forming tool has been worn out, the remaining surface layer can be removed and the whole technological cycle can be repeated.

Industrial applicability

[0019] The method according to the invention can be used for a wide spectrum of forming tools, in particular for forging dies, pull broaches an forging jaws. The technology is extensively applicable to a wide range of tool steel grades, for which it has been developed, such for the grades 38CrMoV5-1, X40CrMoV5-1, or 56CrNiMoV7, and many other ones.

[0020] The applicant's enterprise uses an extended range of hot work tool steels having similar chemical compositions and being suitable for diverse steel forming processes.

[0021] The diagram shown in Fig. 3 illustrates the results of the respective field tests wherein an evident improvement of the tool life values has been achieved. Furthermore, other spin forming tools, which have been treated using the method according to the invention, are undergoing similar tests.

Claims

1. Method of combined surface treatment of tool steels, **characterized in that** the functional portions of a tool undergo laser quenching followed by plasma nitriding in the subsequent step.

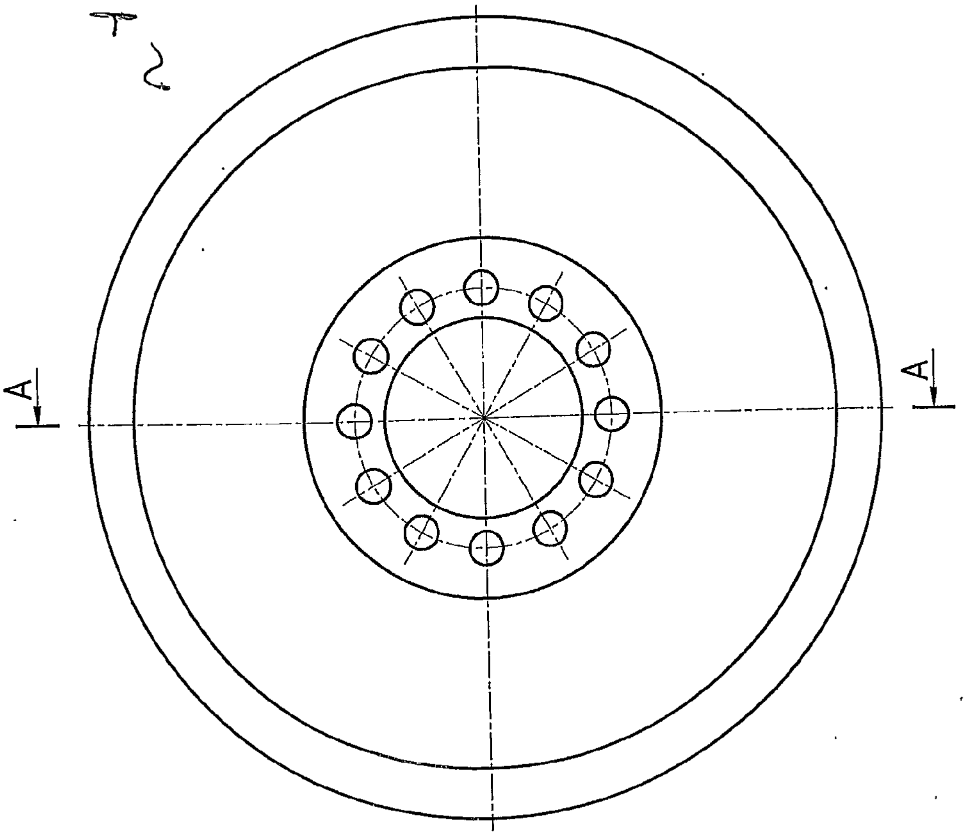

FIG.1

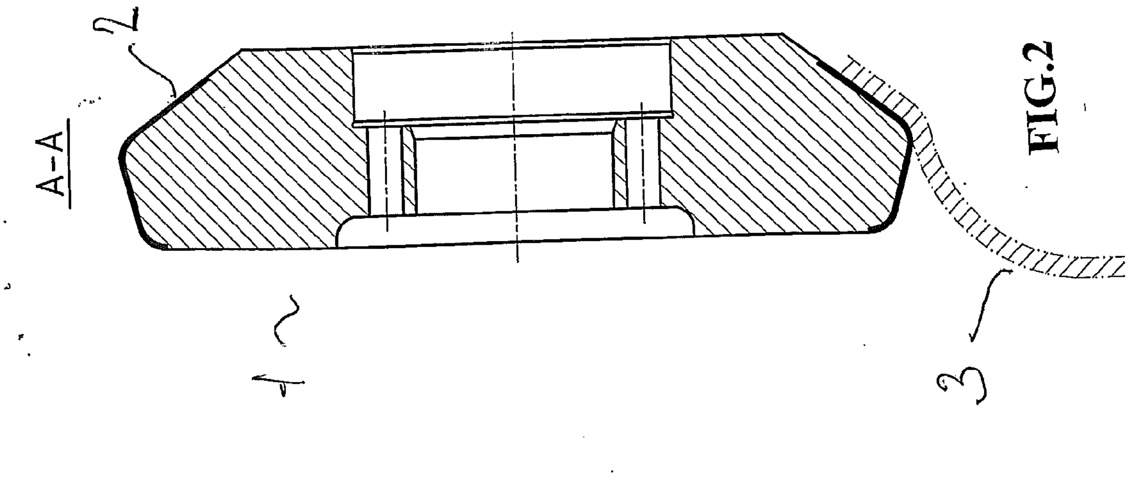

FIG.2

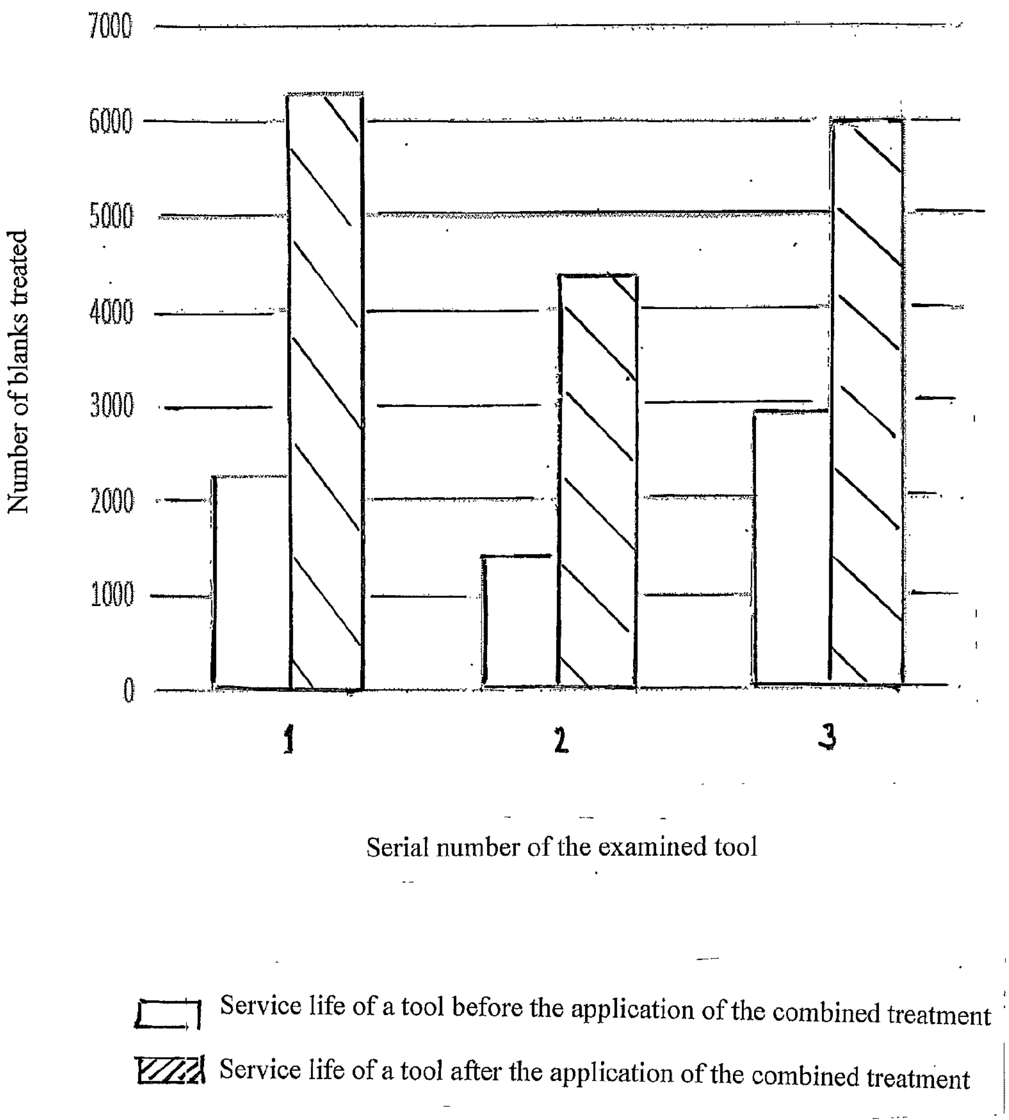
Number of blanks treated
7000
6000
5000
4000
3000
2000
1000
0
1
2
3
Serial number of the examined tool
Service life of a tool before the application of the combined treatment
Service life of a tool after the application of the combined treatment

FIG.3

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EUROPEAN SEARCH REPORT

Application Number
EP 15 15 3854

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	DE 296 22 682 U1 (FERROMATIK MILACRON MASCHINENB [DE]) 3 April 1997 (1997-04-03) * page 1, paragraph 2 * * page 2, last paragraph - page 3, paragraph 4 * * page 4, paragraph 6 - page 5, paragraph 1; claims 1, 2, 6 * -----	1	INV. C23C8/02 C23C8/38 C21D1/09 C23F17/00 C21D6/00
A	CHENG Y ET AL: "Research of the wear resistance of 35CrMoA steel in laser quenching /nitrided layer", PROCEEDINGS OF 2011 INTERNATIONAL CONFERENCE ON ELECTRONIC & MECHANICAL ENGINEERING AND INFORMATION TECHNOLOGY (EMEIT), 12-14 AUGUST 2011, HARBIN, HEILONGJIANG [CN], vol. 7, 12 August 2011 (2011-08-12), pages 3457-3460, XP055200204, DOI: 10.1109/EMEIT.2011.6023067 ISBN: 978-1-61-284087-1 * the whole document * -----	1	
			TECHNICAL FIELDS SEARCHED (IPC)
A	BONSS S: "Harte Linie - Laserstrahlhärten -Integration in die Fertigung ermöglicht schlanke Prozesse", MO METALLOBERFLÄCHE, vol. 61, no. 9, September 2007 (2007-09), pages 34-36, XP001506213, IGT INFORMATIONSGESELLSCHAFT TECHNIK, MÜNCHEN [DE] ISSN: 0026-0797 * the whole document * -----	1	C23C C21D C23F

The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
Munich	6 July 2015	Hoyer, Wolfgang

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE 29622682 U1	03-04-1997	NONE	

EPO FORM P0459

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- EP 0130749 B1 **[0006]**
- GB 202838 A **[0006]**
- WO 9628574 A **[0006]**
- JP 2013234370 A **[0008]**
- KR 100661130 B1 **[0008]**
- US 5536549 B1 **[0008]**