Abstract: This invention relates to the fabrication of jet forming nozzles that can be used, for instance, for the cleaning of surfaces, removal of coatings, production of roughness on surfaces, cutting and separation of materials. The technical result of this invention is increasing the service life of the jet forming nozzle due to a higher wear resistance of the working orifice walls of the nozzle and retaining the compactness of the abrasive jet for a longer time in order to increase the efficiency of the jet forming nozzle for gas and hydraulic machining of materials. Said technical result of this invention is achieved as follows. The jet forming nozzle fabrication method comprises producing a working orifice in a composite diamond material synthesized at high pressures and temperatures using a binder and the final machining of the working orifice followed by nitride hardening. Said composite diamond material comprises a diamond phase and a binder phase comprising nickel and titanium wherein the titanium content is 5.0-15.0 wt.\%.
Jet Forming Nozzle Fabrication Method

Field of the Invention. This invention relates to the fabrication of jet forming nozzles that can be used, for instance, for the cleaning of surfaces, removal of coatings, production of roughness on surfaces, cutting and separation of materials.

Background Art. Jets are typically formed by feeding water or gas at a high pressure to a small orifice nozzle. Various abrasive additives can be introduced into the jet depending on machining type. Precision and efficient machining of materials requires nozzles capable of forming compact and well focused jets and preserving their compactness for a long time. During the passage of an air or gas jet, a near-wall turbulent layer forms on the walls of the working orifice of the nozzle that has a decelerating effect and disturbs the parallel structure of the flow. Therefore the formation of a compact jet to a large extent depends on the machining quality of working orifice. Furthermore, the abrasive impact of the jet on the working orifice walls of the nozzle cause jet abrasion of the walls which acquire traces of wear that lead to the loss of jet compactness and head and an increase in impedance forces. Then the nozzle should be replaced. One should also bear in mind that the nozzle is an expensive component of jet forming devices, and therefore frequent nozzle replacement raises the cost of jet machining processes.

Obviously, the wear resistance of nozzles primarily depends on the integral properties of the material the nozzle is made from as well as on the type of machining used for nozzle fabrication.

Nozzles are fabricated using different superhard polycrystalline materials containing diamond powders and binders. These materials have high hardness, low
friction coefficient due to the high content of diamond powders, high heat conductivity, high wear resistance and hence quite long service life.

Known is polycrystalline diamond synthesis method (RU 1029555, publ. 20.07.1999) comprising exposure of a hollowed piece from a carbon containing material with a catalyst metal located inside to ultrahigh pressure and a high temperature. The abrasive resistance and crystal size of the material are increased by additionally introducing a refractory material in a quantity of 4-90% of the carbon containing material volume. Said refractory material is selected from the group containing titanium, hafnium, vanadium, zirconium, niobium, molybdenum, tantalum, tungsten, rhenium, chromium, their alloys or compounds with carbon, nitrogen or silicon, graphite-like boron nitride and aluminum oxide.

The disadvantage of said known material is its practical unsuitability for the fabrication of nozzle tools due to the large diameter of the composite defect area (2.5 - 4 mm) compared to the tool nozzle diameter (0.2 - 1 mm).

Known is a heat resistant composite sintered diamond material (RU 2312844, publ. 2003) that can be used for the fabrication of cutting tools and as a wear resistant material exposed to friction. Said material is obtained at high pressures ($P = 7.7$ GPa) and high temperatures ($T = 2100$ °C) by sintering diamond powders with a grain size of less than 200 nm without any sintering additives. Said material can be formed to have any configuration by laser or spark erosion machining, grinding and polishing. As the material does not contain binders, it has a high Vickers hardness of 85 GPa and a high wear resistance.

The disadvantage of said known material is the relatively high pressures and very high temperatures required for its synthesis. The equipment capable of developing these conditions does not allow fabricating large pieces, and therefore the material has a narrow application range.
Known is a nozzle fabrication method (US 2002142709, publ. 2002) wherein nozzles are fabricated from a wear resistant sintered polycrystalline diamond material synthesized at high pressures and temperatures. Said material is synthesized by sintering diamond powders in the presence of a catalyst. Said catalyst can either be in the form of a powder mixed with diamond powder or introduced into the diamond powder layer by impregnation. Said binding catalyst can be cobalt or a cobalt alloy, e.g. with nickel. Said material has sufficient wear resistance and electrical conductivity. The sintered diamond material is spark erosion machined or laser cut, or machined using another method suitable for achieving the required shape. The working orifice is produced in the fabricated piece by spark drilling and then ground and polished.

The disadvantage of said known method is as follows. The structure of the sintered diamond material consists of diamond grains bound together with the binder the physical and mechanical properties of which are significantly inferior to those of diamond. During nozzle operation, its working orifice walls are exposed to extensive wear due to the impact of the abrasive jets passing through said orifice at a high pressure. The wall erosion will occur selectively across the surface in the areas where the metallic phase is present to form the so-called erosion pitting. The presence of erosion pitting on the surface of the working orifice disturbs the rectilinear structure of the jet and produces turbulence in the water or gas jet, and as a result the compactness and velocity of the jet decrease, and energy loss increases. This all reduces the efficiency of jet machining.

The prototype of this invention is the method of jet forming nozzle fabrica-
tion (WO 2008032272, publ. 2008) from polycrystalline diamond material comprising cobalt, silicon and silicon carbide as a binder, using an ultrahigh pressure synthesis device. A working orifice is produced in the polycrystalline diamond piece using laser cutting following which said orifice is machined to the required
size and polished to the final size and surface quality. The material is preferably polished with a copper or steel wire in the presence of fine-grained diamond powder.

The disadvantage of said method is the presence of silicon which increases in volume during crystallization, produces microstresses and eventually causes microcracking. During the fabrication of nozzles or other type of tools, the loads produced during machining cause cracking and damage the polycrystalline materials.

**Disclosure of the Invention.** The technical objective of this invention is to provide a jet forming nozzle fabrication method wherein the service life of the jet forming nozzle is increased due to a higher wear resistance of the working orifice walls of the nozzle and the compactness of the abrasive jet is retained for a longer time in order to increase the efficiency of the jet forming nozzle for gas and hydraulic machining of materials.

Said technical result of this invention is achieved as follows.

The jet forming nozzle fabrication method comprises producing a working orifice in a composite diamond material synthesized at high pressures and temperatures using a binder and the final machining of the working orifice followed by nitride hardening.

Said composite diamond material comprises a diamond phase and a binder phase comprising nickel and titanium wherein the titanium content is 5.0 - 15.0 wt.%.

In some embodiments nitride hardening is achieved in a nitrogen containing environment at 700 - 900°C for 1 - 15 h.

In yet another embodiment nitride hardening is achieved in a pure nitrogen environment.
In still another embodiment said diamond material is synthesized from a carbon containing material in the thermodynamic stability region of diamond in the presence of a binder.

In one more embodiment said binder contains 5.0 - 10.0 wt.% titanium.

Alternatively, said composite diamond material is synthesized by sintering diamond powders in the presence of a binder.

In yet another embodiment said composite diamond material is synthesized by impregnation of diamond powders with binder melt.

**Embodiments of the Invention.** The method is implemented as follows.

Nozzles are fabricated from polycrystalline diamond materials produced using different technologies. One method of synthesizing said polycrystalline diamond material comprises mixing of diamond powder with catalyst metal powders and placing the mixture in a device where high pressure and temperature are produced to achieve the catalytic reactions and binding of diamond particles with one another.

Another method of producing said polycrystalline diamond material comprises placing diamond powder in a mold and compacting said powder using any known method e.g. pressing, vibration compacting etc.. A material with a high volume concentration of diamond powder can be synthesized from diamond powders with different grain sizes wherein the grain size of the powders is selected such that powders with smaller grain sizes are located between diamond powders with larger grain sizes. An impregnating material is placed on the compacted powders, and compaction is performed at pressures and temperatures providing for the required flow of the impregnating material.

Alternatively, polycrystalline diamond material can be produced by forming a piece of a carbon containing material that is impregnated with a catalyst metal or alloy at high pressures and temperatures. For this method, the catalyst metal is in
the form of a rod that contacts with said carbon containing material. Impregnation is carried out at pressures of above 7 GPa and a temperature providing for the flowing of the catalyst and its complete penetration into the bulk of the graphite piece. The impregnation temperature depends on catalyst composition. Said catalyst is nickel or its alloys with metals e.g. titanium, molybdenum or chromium. Said carbon containing material is graphite. The conditions of synthesis provide for the formation of diamond that starts at the contact surface between said carbon containing material and develops in depth of said carbon containing material of the carbonado or ballas type.

In all of the abovementioned embodiments said composite diamond material comprises a diamond phase and a binder phase, the latter comprising nickel and titanium with the titanium content being 5.0 - 15.0 wt.%.

The nickel/titanium alloy has high strength and satisfactory plasticity. Excess of titanium causes embrittlement of the matrix and hence reduces the strength of the whole composite material. Insufficient titanium content complicates further nitride hardening.

Nozzles are fabricated by forming a working orifice in the polycrystalline piece. As the polycrystalline material with a nickel binder is electrically conductive, the working orifice can be fabricated using spark machining, laser cutting or any other known suitable method.

Orifice production is followed by its multistage machining. First the orifice is rough ground using a wire or needle shaped tool in the presence of coarse grained diamond powder. Then the orifice is ground with fine grained diamond powder.

The orifice is polished to a smooth surface capable of forming a compact gas or hydraulic abrasive jet with a constant pressure that flows from the working orifice without breaking or spraying.
After the final machining the working orifice is nitride hardened. Nitride hardening of the entire nozzle is more technically convenient. Nitride hardening causes diffusion saturation of metal containing regions. These regions are metal matrix that comprises metals or catalyst alloys. As the main nozzle material is polycrystalline diamond comprising a nickel/titanium alloy binder, the surface of the orifice walls comprises regions of that alloy.

These regions are diffusion saturated to form a nitride layer. Nitride layer formation increases the hardness and wear resistance of these regions thus reducing the difference in the physical and mechanical properties between the binder regions and the diamond material regions. The process causes a general increase in the wear resistance of the working orifice walls, their stability to burring and cavitation, and increase the corrosion resistance of the nozzle in water and air media.

For nitride hardening the nozzle is placed into a furnace, nitrogen gas is fed and the furnace is heated to 700-900 °C. As the nozzles have small sizes, chamber furnaces can be used for nitride hardening.

Pure nitrogen is preferably used for nitride hardening. Alternatively, ammonia or ammonia/nitrogen mixture can be used, but in that case the nitride layer with be more brittle.

Nozzles are exposed to the nitrogen atmosphere for 1 - 15 h. At the above-mentioned temperature, a 1-15 h exposure produces a 35 - 40 nm thick nitride layer on the working orifice wall surface. The nitride layer of said thickness does not modify the working channel wall surface relief, retains its smoothness and does not exfoliate from the surface.

 Longer time of nitride hardening will increase the coating thickness and cause coating exfoliation from the matrix due to the difference in the thermal expansion coefficients of the materials, partially depending on diamond content in the nitride hardened surface. Shorter time of nitride hardening will reduce the layer
thickness. If the layer thickness is insufficient, the required continuity and density cannot be achieved.

Increasing the processing temperature to above 900°C will increase the rate of nitride hardening but will also cause graphitization of diamond grains in the compacted material and reduce the abrasion resistance of the material. Reducing the processing temperature to below 700°C will largely increase the required nitride hardening time and result in an unjustified reduction in nitride hardening process output.

Example.

Several diamond composite materials were produced by synthesizing from graphite at 0.9 GPa and approx. 1700°C for 10 s. An alloy containing nickel and 10% titanium was used as the catalyst. The composite material pieces were 4 mm in diameter and 4 mm in height. The diamond composite material pieces were ground to 2 mm thick and 4 mm diameter plates. From these plates, 2 mm outer diameter and 0.3 mm orifice diameter nozzles were cut using laser cutting. The orifices were then machined and polished to achieve a roughness of Ra = 0.32. Nitride hardening was carried out at a 1.2 - 1.5 atm pressure and 850°C for 3 h. The nitride coating thickness on the metallic regions of the diamond composite material surface reached 30-40 nm. The surface roughness did not change compared to the initial as-polished one. For other polycrystalline pieces, the nozzles were nitride hardened at 700°C. The nitride hardening time required for the achievement of dense and continuous coatings was 15 h. Further reduction of the nitride hardening was not efficient because significant time would then be required to obtain a 30-40 nm nitride layer.

Diamond composite materials were also produced using an alloy containing nickel and 20% titanium as the catalyst. High titanium content caused embrittle-
ment of the polycrystalline material, and the strength of the nozzles was significantly lower.

Composite materials synthesized using an alloy containing nickel and 5% titanium as the catalyst retained their integrity and were used for the fabrication of nozzles. The nitride hardening time required for the achievement of a dense nitride layer was 15 h. Reduction of the titanium content to below 5 wt.% complicated nitride hardening, while the increase in the wear resistance of those nozzles was but little.

The nozzles fabricated in accordance with this invention were tested by water abrasion cutting of 20 mm steel sheets at a water pressure of 3800 atm. The compactness of the jets was assessed visually. The jets produced by nitride hardened nozzles did not exhibit overspraying. The cutting capacity of the water abrasion jet was assessed based on steel plate cutting rate. The nitride hardened nozzles provided for a 20% higher cutting rate compared with not nitride hardened ones, and the resistance of the nitride hardened nozzles was 40-60% higher than that of not nitride hardened ones.
What is claimed is a

1. Jet forming nozzle fabrication method comprises producing a working orifice in a composite diamond material synthesized at high pressures and temperatures using a binder and the final machining of the working orifice followed by nitride hardening, wherein said composite diamond material comprises a diamond phase and a binder phase comprising nickel or titanium wherein the titanium content is 5.0 - 15.0 wt.%.

2. Method of Claim 1 wherein nitride hardening is achieved in a nitrogen containing environment at 700 - 900°C for 1 - 15 h.

3. Method of Claim 2 wherein nitride hardening is achieved in a pure nitrogen environment.

4. Method of Claim 1 wherein said diamond material is synthesized from a carbon containing material in the thermodynamic stability region of diamond in the presence of a binder.

5. Method of Claim 4 wherein said binder contains 5.0 - 10.0 wt.% titanium.

6. Method of Claim 1 wherein said composite diamond material is synthesized by sintering diamond powders in the presence of a binder.

7. Method of Claim 1 wherein said composite diamond material is synthesized by impregnation of diamond powders with binder melt.
**A. CLASSIFICATION OF SUBJECT MATTER**

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**B. FIELDS SEARCHED**

- C01B 3 1/00, 3 1/06, C04B 35/50, 35/52, 35/78, B24C 5/00, 5/04, B24D 3/00, 3/06

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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**Further documents are listed in the continuation of Box C.**

**See patent family annex.**

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**Date of the actual completion of the international search**

12 August 2013 (12.08.2013)

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