

Selected Patents Related to Thermal Spraying

Issued between April 1, 2007
and June 30, 2007

Prepared by Jiří Matějčík, Institute of Plasma Physics, Za Slovankou 3, 18200 Praha 8, Czech Republic; jmatejic@ipp.cas.cz; tel: +420-266 053 307. Adapted with permission from Delphion, <http://www.delphion.com/>.

CA denotes Canadian patent, DE denotes German patent, EP denotes European patent, KR denotes Korean patent, RU denotes Russian patent, US denotes United States patent, WO denotes World Intellectual Property Organization application. The information has the following format: Title, Abstract, Patent number, Inventors, Company, Issued/Filed dates.

Applications

Ceramic Hardfacing for Progressing Cavity Pump Rotors. A hardfacing for downhole progressing cavity pumps is disclosed as well as a method for producing same. The hardfacing consists of a ceramic layer applied to a ferrous pump rotor body by way of plasma spraying and a top layer of metallic material having a lower hardness than the ceramic. The ceramic layer has a grainy surface with a plurality of peaks and intermediate depressions, the peaks being formed by ceramic grains at the surface of the ceramic layer. The thickness of the top layer is adjusted such that the depressions between the peaks of the ceramic layer are completely filled thereby providing the rotor with a ceramic hardfacing of significantly reduced surface roughness. In the process of the invention, the pump rotor, which may be provided with a molybdenum bonding layer, is plasma coated with the ceramic, and the resulting ceramic layer is covered with the metallic material top layer. The top layer is polished either until the dimensions thereof are within the tolerances acceptable for the finished rotor or until a majority of the peaks of the ceramic layer are exposed.

CA 2337622: R.A.R. Mills. Company: Kudu Industries, Inc. Issued/Filed: May 1, 2007/February 21, 2001.

Coating Head. It is intended to reduce the possibility of flanging on a coating head of coating applicator used to apply

a coating liquid onto a surface of coating object member, such as a sheetlike member. There is provided a coating head having a member of sintered hard alloy effectively arranged in a distal end portion of the coating head, in which the above member is assembled to avoid any interstice permitting penetration of coating material. The coating head can be characterized in that its distal end portion is coated with a sintered hard alloy with specified thickness. This sintered hard alloy may be a cermet containing tungsten carbide (WC). Further, this coating can be performed by thermal spraying.

WO 27049349: S. Touoka and Y. Nagata. Company: Hirata Corp., Crystal Optics Inc. Issued/Filed: May 3, 2007/October 27, 2005.

Copper Thermal Spray Coating of the Building Interior. KR 708977: S.U. Jo and S.H. Jung. Issued/Filed: April 11, 2007/September 16, 2006.

Die Coatings for Gravity and Low-Pressure Die Casting. A die coating for use on the surface of a metal mold or die component contacted by molten metal in low-pressure or gravity die casting, and a method for its production. The die coating includes a porous layer of ceramic material produced by codeposition, using a thermal spraying procedure, of a powder of said ceramic material, and a powder of an organic polymer material. After the codeposition, the codeposited layer is heated to remove the polymer material and provide the porous layer of ceramic material.

US 7234507: M. Jahedi, M. Giannos, and S. Gulizia. Company: Cast Centre Pty Ltd., Department of Mining, Minerals and Materials, Engineering, University of Queensland. Issued/Filed: June 26, 2007/January 2, 2004.

Fabrication of Electrode Structures by Thermal Spraying. A method for the rapid production of electrode structures such as Cu-SDC anodes for use in direct oxidation solid oxide fuel cells involves codepositing a copper-containing material and a ceramic by plasma spraying to form a coating on a substrate. Layers of CuO-SDC have been codeposited by air plasma spraying, followed by in situ reduction of the

CuO to Cu in the anodes. Materials having catalytic properties, such as cobalt, may also be incorporated in the structures. Controlled compositional or microstructural gradients may be applied to optimize the microstructure and composition of the coatings.

WO 27048253: O. Kesler and N. Ben-Oved. Company: The University of British Columbia. Issued/Filed: May 3, 2007/October 27, 2006.

Improvements in Thermal Sprayed Tooling. A metallic shell used, for example as a mold, is formed by spray deposition connected to a base by rods or other supports connected to mounting elements that are incorporated in the shell during the deposition process. The shell can incorporate different metal to provide different thermal conductivity in various regions.

CA 2408703: C.P. Covino. Company: GMIC, Corp. Issued/Filed: June 19, 2007/May 22, 2001.

Method for Coating a Component. The invention relates to a method for coating a component consisting of a fiber-reinforced composite material. According to said method: (a) a composite consisting of organic and metallic parts is applied to a surface of the component to be coated, as an adhesive layer, by means of thermal spraying, (b) a layer predominantly comprising metallic parts is applied to the adhesive layer, as an intermediate layer, by means of thermal or kinetic spraying, and (c) a functional covering layer consisting of metal, a metal-carbide composite, oxide ceramics, or mixtures of said materials is applied to the intermediate layer by means of thermal or kinetic spraying.

WO 27045217: T. Stoltenhoff and K. Gorris. Company: Praxair Surface Technologies GmbH. Issued/Filed: April 26, 2007/October 12, 2006.

Methods of Making Al₂O₃-SiO₂ Ceramics. Methods for making glasses and glass-ceramics comprising Al₂O₃ and SiO₂. Glasses made according to the present invention can be made, formed as, or converted into glass beads, articles (e.g., plates), fibers, particles, and thin coatings. Some embodiments of glass-ceramic particles made

according to the present invention can be particularly useful as abrasive particles.

US 7197896: A. Celikkaya and T.J. Anderson. Company: 3 M Innovative Properties Co. Issued/Filed: April 3, 2007/Sept 5, 2003.

Method for Manufacturing Molds and Dies by Thermal Spraying. KR 723126: B.G. Seong, J.H. Kim, J.H. Ahn, and S.R. Oh. Company: Posco, Research Institute of Industrial Science & Technology. Issued/Filed: May 22, 2007/Dec 24, 2005.

Plasma Sprayed Layer on Cylinder Bores of Engine Blocks. Powder contains (in wt.%) 0.4-1.5 C, 0.2-2.5 Cr, 0.2-3 Mn, and a balance of iron. Independent claims are also included for the following: iron-containing layer applied by plasma spraying. Process for producing the iron-containing layer.

EP 1507020: G. Barbezat. Company: Sulzer Metco AG. Issued/Filed: June 27, 2007/Dec 8, 1999.

Plasma Torch with Corrosive Protected Collimator. To protect the collimator of a transferred plasma arc torch from premature failure due to corrosion, an anticorrosive covering is applied on the exposed face surface and a portion of the inner exit bore of the collimator. The specification describes several methods for producing the collimator for a plasma torch having an anticorrosive coating or cladding on the exposed surfaces thereof, including electroplating, electroless plating, flame spraying, plasma spraying, plasma transferred arc, hot isostatic pressing, and explosive cladding.

WO 27040583: G.J. Hanus, R.E. Reeve, and T.J. Stahl. Company: Phoenix Solutions Co. Issued/Filed: April 12, 2007/Feb 14, 2006.

Solid Seal Which Is Obtained by Means of Thermal Spraying. The invention relates to an element that is made from a composite material comprising a microcracked matrix that takes the form of a three-dimensional interconnected network of microcracks and that is open at the surface of the ceramic matrix. According to the invention, an additive material, consisting of a flux or glass, is dispersed in the matrix, said additive material comprising a material that, when the composite material is brought to a determined temperature,

softens, and migrates by capillary action through the network of microcracks to the surface of the element. A sufficient quantity of additive material is dispersed in the matrix in relation to same in order to coat an open surface of the composite element such as to create a gas-tight barrier thereon.

WO 27042505: L. Bianchi, J. Toulchoat, and C. Bories. Company: Commissariat a l'energie atomique. Issued/Filed: April 19, 2007/Oct 9, 2006.

Sound-Proofing Component and Method for the Production Thereof. The invention relates to a sound-proofing component, particularly for absorbing noise in motor vehicles, comprising a first layer that is made of a nonwoven material, is embodied in a planar manner similar to a mat, and is provided with two opposite surfaces. A thin spray coating made of a duroplast, such as polyurethane and/or thermoplastic materials, is disposed on one of the outer surfaces of the nonwoven fabric. Also disclosed is a method for producing said component.

WO 27054346: O. Hessler. Company: Ideal Automotive GmbH. Issued/Filed: May 18, 2007/Nov 10, 2006.

Stepped Gradient Fuel Electrode and Method for Making the Same. The present invention provides a method of depositing a stepped-gradient fuel electrode onto a fuel cell support and the resulting fuel cell, that comprises placing a solid oxide fuel cell support that has at least an air electrode layer and an electrolyte layer into an atmospheric plasma spraying chamber and measuring spray parameters of an atmospheric plasma spray to obtain reactive oxides, conductive metal, and graphite phases. Then spraying the spray parameters onto the solid oxide fuel cell support to produce multiple sublayers on the solid oxide fuel cell support, and adjusting usage of the atmospheric plasma spray. The adjusting of the hydrogen usage comprises using high hydrogen levels for the initial spraying of the sublayers producing a first gradient region, and as lower hydrogen level for subsequent spraying of the sublayers, producing a second gradient region.

WO 27067242: K. Huang, H.D. Harter, and P.G. Turkal. Company: Siemens Power Generation, Inc. Issued/Filed: June 14, 2007/Sept 20, 2006.

Structures and Methods for Damping Tool Waves Particularly for Acoustic Logging Tools. A toner donor roll for use in a development apparatus of an electrophotographic apparatus is disclosed. The donor roll includes a conductive core of a ceramic outer coating over the conductive core, the ceramic coating formed from thermal spraying a single homogeneous powder consisting of particles each of which contains a specific ratio of pure alumina and pure titania held together with an organic binder.

CA 2410168: J.B. Aron, H.C. Straub, C. Esmersoy, D. Grigor, R.M. D'Angelo, R.D. Joyce, J.A. Pabon, C.-J. Hsu, L. Reid, and P. Campanac. Company: Schlumberger Canada Ltd. Issued/Filed: June 26, 2007/Oct 29, 2002.

Thermal Spray Coated Rolls. This invention relates to rolls for use in or in contact with molten metal comprising a roll drum having an outer peripheral surface and a thermally sprayed coating on the outer peripheral surface of said roll drum, said thermally sprayed coating comprising from about 66 to about 88 wt.% W, from about 2.5 to about 6 wt.% C, from about 6 to about 20 wt.% Co, and from about 2 to about 9 wt.% Cr; a process for preparing the rolls; a method for forming a metal layer on a metal sheet using the rolls, e.g., galvanization; and a thermal spray powder for coating the outer peripheral surface of the rolls.

WO 27047330: W. Jarosinski, J. Quest, D. Wang, V. Belov, and A.S. Kleyman. Company: Praxair S.T. Technology, Inc. Issued/Filed: April 26, 2007/Oct 12, 2006.

Diagnostics and Characterization

Methods for Preparing and Testing a Thermal Spray Coated Substrate. A method for fabricating and testing an article having a thermal spray coating thereon. The method includes providing a substrate article having a surface, thermally spraying a coating material onto the surface of the substrate article, wherein a surface of contact between the coating material and the substrate article is a bond line, and nondestructively testing the coated article. Non-destructive testing includes generating an eddy current in the coated article, measuring the eddy current in the coated article, and evaluating a

near-bond-line region of the coated article located adjacent to the bond line using the measured eddy current.

US 7229661: P.A. Ruzzo, M. Stewart, and A.W. Mellors. Company: General Electric Co. Issued/Filed: June 12, 2007/Sept 1, 2006.

Feedstock

Ceramic Powders and Thermal Barrier Coatings. This invention relates to ceramic powders comprising a zirconia-based component, e.g., yttria-stabilized zirconia, and an (alumina + silica) based component, e.g., mullite. The ceramic powders are useful for forming thermal shock resistant coatings having the same composition, through deposition by thermal spray devices. This invention also relates to thermal barrier coating systems suitable for protecting components exposed to high-temperature environments, such as the thermal environment of a gas turbine engine. This invention further relates to forming free-standing solid ceramic articles.

WO 27053493: T.A. Taylor. Company: Praxair S.T. Technology, Inc. Issued/Filed: May 10, 2007/Oct 30, 2006.

Corrosion-Resistant Powder and Coating. The invention is a corrosion-resistant powder useful for deposition through thermal spray devices. The powder consists essentially of, by weight percent, 30-60 W, 27-60 Cr, 1.5-6 C, a total of 10-40 Co plus Ni and incidental impurities plus melting point suppressants.

CA 2477853: W.J.C. Jarosinski and L.B. Temples. Company: Praxair S.T. Technology, Inc. Issued/Filed: May 1, 2007/Feb 19, 2003.

Plasma Spheroidized Ceramic Powder. Invention provides preparation of chemically homogeneous powder suitable for thermal spraying. Zirconium dioxide is first subjected to electric fusion using up to 60 wt.% oxide appropriate to stabilize zirconium dioxide in tetragonal phase followed by sharp cooling of thus obtained stabilized zirconium dioxide and heat treatment to form mainly spherical hollow particles of stabilized zirconium dioxide 200 μm or less in size. Powder suitable for applying thermal barrier forming coating onto a substrate contains morphologically and chemically uniform

stabilized zirconium dioxide including spheroidized hollow particles. Effect: optimized preparation process.

RU 2299926: H. Wallar. Company: Saint-Gobain Ceramics And Plastics, Inc. Issued/Filed: May 27, 2007/Aug 4, 2003.

Thermally Sprayed Powder and the Method of Its Production. The invention pertains to the method of production of the thermally sprayed powders and may be used in production of the powders out of the chromium dioxide for the thermally sprayed wear-resistant coatings. The thermally sprayed powder contains from 45 up to 99 mass% chromium dioxide and from 1 up to 55 mass% alpha aluminum oxide, and less than 50 shares per one million of the alkaline metals and the alkaline earth metals stabilizing chromium in its hexavalent state. The powder particles have primarily the single-phase crystalline structure with the aluminum oxide share in other phases, except for the alpha phase, of no more than 10 mass% of the full contents of the aluminum oxide. The method of production of the thermally sprayed powder includes: stirring action of the aluminum oxide powder containing the impurities of the alkaline metals and the alkaline earth metals of no more than 120 shares per one million of each element with the chromium dioxide powder, which also has less than 120 shares per one million of the impurities of the alkaline metals and the alkaline earth metals stabilizing chromium in the hexavalent state; burning of the mixture at the temperature of 1300-1500 $^{\circ}\text{C}$. The invention allows reduction of formation of the toxic compounds of the hexavalent chromium during the thermal spraying of the wear-resistant powders of chromium dioxide. Effect: the invention ensures the reduced formation of the toxic compounds of the hexavalent chromium during the thermal spraying of the wear-resistant powders of chromium dioxide.

RU 2298527: S.H. Ju and H. Wallar. Company: Saint-Gobain Ceramics And Plastics, Inc. Issued/Filed: May 10, 2007/April 4, 2003.

Spraying Systems and Methods

Apparatus for Thermal Spray Coating. A system for thermal spray coating of a particulate material onto a substrate

includes a spray gun apparatus having dual vortex chambers for the mixing of fuel gas and oxygen. The apparatus provides a jet flame resulting from a compression wave formed by compressed air. Dual venturis control the flow of fluidized coating material particles to provide smooth and controlled delivery of coating material to the spray gun.

US 7216814: T. Gardega. Company: Xiom Corp. Issued/Filed: May 15, 2007/July 30, 2004.

Electric Arc Spraying System. An electric arc spraying system includes a spraying gun for thermally spraying an inner surface of an object such as a cylinder block by blasting compressed gas substantially perpendicularly to the supplying direction of target wires. The spraying gun is rotated by a spraying gun rotation mechanism. The target wires are loaded in and supplied from wire supplying sources. A wire feeder rotation mechanism is provided for rotating the wire supplying sources synchronously with the spraying gun in rotation. Wire feeders are provided at the spraying gun or adjacent to the wire supplying sources for feeding the target wires. Wire support cables are configured to guide the target wires from the wire supplying sources to the spraying gun.

US 7210638: G. Tujii, Y. Nakamura, M. Uchida, K. Kodama, N. Kondo, and N. Miyamoto. Company: Daihen Corp., Toyota Jidosha Kabushiki Kaisha. Issued/Filed: May 1, 2007/Sept 21, 2006.

Improved Plasma Spraying Method and Apparatus. A method of depositing a coating of spray particles from a coating material feed onto a target substrate. A plasma arc is provided in a gas through which a plasma arc current passes. The plasma arc current is modulated by plasma arc current pulses having a frequency and a magnitude. The coating is provided by injecting the coating material into the plasma arc from a coating material feed. The deposition rate of spray coating is measured. The stand-off distance between the target substrate and the coating material feed is adjusted to provide maximum deposition rate. Other features of the spray process can also be measured and adjusted.

DE 19881726: B. Goodman. Company: Plasma Model Ltd. Issued/Filed: April 19, 2007/Oct 19, 1998.

Installation for the Plasma Spraying on the Crankshafts. The invention pertains to the installation of the plasma spraying on the crankshafts and may find application in the mechanical engineering. The installation has the heat-sound-insulation chamber, which consists of the shot-blasting section and the spraying section separated by the septum. The gear of transportation of the details consists of: the stationary guides, the movable guide with the bracket, the plate for mounting of the motor-reducer, the pneumatic cylinder for transportation of the detail into the spraying section and the pneumatic cylinders for transportation of the details into the shot-blasting section. The stationary guides are placed on the cover of the heat-sound-insulation chamber perpendicularly to the detail rotation axis. The movable guide is connected to the stationary guides and on it there is the motor-reducer mounting plate linked with the pneumatic cylinder rod used for transportation of the detail into the spraying section. This rod is rigidly fixed on the movable guide bracket. The indicated movable guide is rigidly connected with the rods of the pneumatic cylinders for transportation of the detail into the shot-blasting section. The rods are rigidly fixed on the cover of the heat-sound-insulation chamber. Such design of the installation allows to increase the adhesion strength of the produced coating due to reduction of the time between the shot-blasting treatment and the spraying and also to decrease the labor input used for the detail mounting at the centers. Effect: the invention ensures the increased adhesion strength of the produced coating, reduction of the time between the shot-blasting treatment and the spraying operations, the decreased labor input used for the detail mounting at the centers.

RU 2300578: A.B. Kobernichenko, V.V. Saltan, V.V. Efremov, R.A. Nagaev, and R.V. Gilevskij. Company: Rjazanskij Voennyj Avtomobil'nyj Institut. Issued/ Filed: June 10, 2007/April 11, 2005.

Plasma Spraying Device. Plasma spraying device for spraying coating powder has electrically conducting tubular lines for feeding coolant, electrical energy with mechanical interfaces for shape- and force-locking connection between burner shaft, burner head lines.

EP 1692922: S. Keller. Company: AMT AG. Issued/Filed: April 25, 2007/Oct 8, 2004.

Plasma Spray Nozzle System. A plasma spray nozzle system for uniform injection of feedstock into a plasma stream. A plasma nozzle is provided, having a bore with an axis, the bore shaped for the passage of a plasma stream. An annular plenum chamber is provided coaxial with and substantially surrounding the bore. A plurality of feedstock injection passages extend from the plenum chamber and converge toward the plasma stream. The plenum chamber is supplied continuously with feedstock material from an outside source. The feedstock is distributed throughout the plenum chamber volume and is exhausted into the plasma stream via the plurality of feedstock injection passages. By selecting an appropriate number of feedstock injection passages, a more uniform and symmetrical loading with feedstock of the plasma stream is achieved.

WO 27065252: L.B. Delcea. Issued/ Filed: June 14, 2007/Nov 27, 2006.

Spray Coating Apparatus and Fixtures. A system for applying a sprayed coating includes a spray mechanism operative to spray a liquefied coating material; a target system including a rotatable spray target wheel; and one or more device-holding fixtures configured to be mounted onto the spray target wheel without requiring either an unattached threaded fastener or a locking pin. Preferred embodiments of the system are configured for thermal spray application of tamper-resistant coatings (TRCs).

US 7208046: C.W. Anderson, L. Reeves, B. Heggli, and Jr. T. W. Dowland. Company: White Electronic Designs Corp. Issued/Filed: April 24, 2007/Jan 10, 2003.

Suspension Plasma Spray Deposition. The plasma spray method produces a material deposit on a substrate to form either a protective coating or a near-net shape body, or produces a powder of a given material. The material is supplied to a plasma discharge in the form of a suspension comprising small solid particles of that material dispersed into a liquid or semiliquid carrier substance. The suspension is brought into the plasma discharge by an

atomizing probe using a pressurized gas to shear the suspension and thus atomize it into a stream of fine droplets. The plasma discharge vaporizes the carrier substance and agglomerate the small solid particles into partially or totally melted drops that are accelerated to hit the substrate and form thereon the coating or near-net shape body. Alternatively, the molten drops can be solidified in-flight and collected into a vessel to produce a powder of that material.

CA 2198622: F. Gitzhofer, M.I. Boulos, and E. Bouyer. Company: Universite de Sherbrooke. Issued/Filed: May 8, 2007/Aug 28, 1995.

Thermal Barrier Coatings and Bond Coats

Structural Environmentally Protective Coating. A coating suitable for use as an environmentally protective coating on surfaces of components used in hostile thermal environments, including the turbine, combustor, and augmentor sections of a gas turbine engine. The coating is used in a coating system deposited on a substrate formed of a superalloy material. The coating contacts a surface of the superalloy substrate and is formed of a coating material having a tensile strength of more than 50% of the superalloy material. The coating material is predominantly at least one metal chosen from the group consisting of platinum, rhodium, palladium, and iridium and has sufficient strength to significantly contribute to the strength of the component on which the coating is deposited.

US 7208232: M.D. Goran and R. Darolia. Company: General Electric Co. Issued/Filed: April 24, 2007/Nov 29, 2005.

Thermal Barrier Coating Containing Reactive Protective Materials and Method for Preparing Same. A thermal barrier coating for an underlying metal substrate of articles that operate at, or are exposed to, high temperatures, as well as being exposed to environmental contaminant compositions. This coating comprises an inner layer nearest to the underlying metal substrate comprising a ceramic thermal barrier coating material, as well as an outer layer having an exposed

surface and comprising a CMAS-reactive material in an amount up to 100% and sufficient to protect the thermal barrier coating at least partially against CMAS that becomes deposited on the exposed surface, the CMAS-reactive material comprising an alkaline earth aluminate or alkaline earth aluminosilicate where the alkaline earth is selected from barium, strontium, and mixtures thereof, and optionally a ceramic thermal barrier coating material. This coating can be used to provide a thermally protected article having a metal substrate and optionally a bond coat layer adjacent to and overlaying the metal substrate. The thermal barrier coating can be prepared by forming the inner layer of the ceramic thermal barrier coating material, followed by depositing the CMAS-reactive material, or codepositing the CMAS-reactive material and the ceramic thermal barrier coating material, to form the outer layer.

US 7226668: B.A. Nagaraj and I. Spitsberg. Company: General Electric Co. Issued/Filed: June 5, 2007/Dec 12, 2002.

Thermal Barrier Coating with Stabilized Compliant Microstructure. A thermal barrier coating for a gas turbine component includes a bond coating layer, at least a first segmented columnar ceramic layer on the bond coating layer, and a particulate structure-stabilizing material disposed within a plurality of segmentation gaps within the columnar ceramic layer(s). The thermal barrier coating may further comprise a second segmented columnar ceramic layer of yttria-stabilized hafnia on the first segmented columnar ceramic layer, and an outer, continuous, nonsegmented sealant layer covering the yttria-stabilized hafnia layer to prevent ingress of extraneous materials into the segmentation gaps. Methods for depositing a thermal barrier coating on a substrate are also disclosed.

WO 27037773: T.E. Strangmann. Company: Honeywell International, Inc. Issued/Filed: April 5, 2007/July 16, 2004.

Thermal Barrier Coating System and Methods. Disclosed is a noble metal bond coat of a thermal barrier coating

system useful for enhancing adhesion of a ceramic topcoat to a superalloy substrate. The bond coat includes about 10-30 wt.% Al, about 2-60 wt.% noble metal, between trace amounts and about 3 wt.% of a reactive element selected from the group consisting of yttrium, zirconium, hafnium, scandium, all the lanthanides, and mixtures thereof, and balance selected from the group consisting of nickel, cobalt, iron, and mixtures thereof, wherein the bond coat is further characterized by absence of added chromium. One method includes plasma spraying a prealloyed powder of the bond coat composition on the substrate followed by alumina formation and ceramic topcoat deposition. Uses include thermal barrier coating systems on gas turbine engine hot section components such as turbine blade and vane airfoils, combustors, and exhaust nozzles.

EP 995817: W. Beele, A.H.F. van Lieshout, G.H. Marijnissen, and D.H. Maxwell. Company: Sulzer Metco Coatings B.V. Issued/Filed: May 16, 2007/Oct 19, 1999.