Chapter 6
New Methods of Laser Micro-Nanomanufacturing

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Laser manufacturing is a manufacturing method that uses the interaction between laser and matter to make it undergo heating, melting, vaporization, evaporation, sublimation, coulomb explosion, electrostatic stripping and other processes, thus realizing the forming and forming of parts/components. The laser manufacturing industry has developed rapidly in the past ten years, especially with the development of aviation, aerospace, high-end chips, new energy, transportation and other fields in China, the demand for laser manufacturing of high-performance components has become more and more urgent. Ultra-fast laser micro-nanomanufacturing is one of the frontiers of laser manufacturing, with the characteristics ultra- fast (<100 fs), super strong (>1, 14 W/cm^2), it has unique advantages in the manufacture of difficult-to-machine materials and three-dimensional complex curved surface

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micro-nano structures. With the continuous development of ultra- fast laser technology, it is expected to become one of the main means of high-end manufacturing in the future, which can provide key manufacturing support for the development of new energy, aerospace, national defense and other fields, and has important strategic significance for promoting China to become a manufacturing power. Ultra-fast laser processing faces the following common challenges in the manufacture of core components in the above fields: the processing materials are diverse and difficult to process, the processing surface is a three-dimensional complex curved surface, the size limit is continuously pushed to new extremes, and the quality requirements are continuously pushed to new extremes. These challenges put forward new requirements for ultra-fast laser manufacturing. It is necessary to understand, observe and control the energy absorption, transfer and corresponding phase transition mechanism in ultra-fast laser processing from the electronic level, and explore the change law and control mechanism of nanoscale electronic dynamic characteristics in the interaction between nonlinear and non-equilibrium ultra-fast laser and materials.

6.1 New Principle and Method of Ultra-Fast Laser Space-Time Shaping Micro-Nano Machining

In the process of ultra-fast laser manufacturing, the absorption of laser energy by materials is mostly completed by electrons as carriers. The pulse width of ultra-fast laser is one thousandth to one hundredth of the heat conduction time (10^{-12}–10^{-10} s) between electron and lattice. Therefore, during femtosecond pulse irradiation, the lattice motion can be ignored, and only the change of electronic state under ultra-fast light field needs to be considered. Although the irradiation time of femtosecond laser pulse to materials is very short (femtosecond order of magnitude), all subsequent processing processes (second order of magnitude) are determined by the interaction between femtosecond laser and electrons, so the local instantaneous electronic state must be regulated [1]. In the past, the observation and regulation of basic manufacturing research were limited to atomic, molecular and above levels. The core scientific problem is whether local instantaneous electronic dynamics can be regulated. The rapid development of ultra-fast laser technology provides feasibility for solving this scientific problem. In the past two decades, ultra-fast laser technology has won 3 Nobel Prizes, resulting in a total of 5 Nobel Prize winners. For example, Professor Ahmed H. Zewail, winner of the Nobel Prize in Chemistry in 1999, has made the world's fastest camera with femtosecond laser as a tool, has observed the process of electron relaxation changes in chemical reactions, and is expected to make breakthroughs in new principles/methods of manufacturing [2].

Inspired by Professor Zewail's work, the research team of Beijing Institute of Technology proposed a new principle of electronic dynamic control processing [1]: by designing the temporal and spatial distribution of ultra-fast laser to adjust the

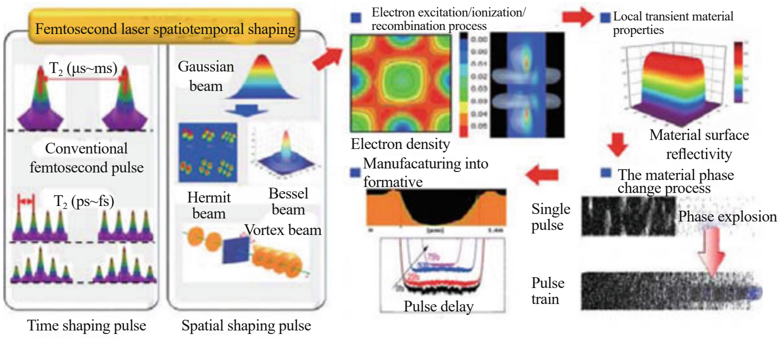

Fig. 6.1 New principle/method of femtosecond laser spatio-temporal shaping

interaction process between laser and electron, active regulation of local instantaneous electronic dynamics (density/temperature/excited state distribution, etc.) is realized, thus regulating the local instantaneous characteristics of materials (reflectivity/refractive index/conductivity, etc.), further regulating the phase transformation, forming and forming processes of materials, and realizing a brand- new target manufacturing method to improve processing quality, efficiency, accuracy and consistency, as shown in Fig. 6.1.

The interaction mechanism between ultra-fast laser and materials is studied by using plasma quantum model, improved two-temperature model, molecular dynamics model and first-principles calculation. It is predicted that the electron excitation/ionization/recombination process, local instantaneous material characteristics, material phase transition process and final forming and forming process can be controlled by ultra-fast laser spatio-temporal shaping [1, 3–5]. The ablation shape and surface texture structure of femtosecond laser were predicted for the first time, and the stable machining depth region with nanometer super diffraction limit was successfully predicted [6–9].

Based on the new principle of electronic dynamic control machining, for femtosecond laser shaping in the time domain, spatial domain, for the first time, the active control of local instantaneous electron dynamics in manufacturing has been realized, greatly improving the processing quality, precision, efficiency, depth-diameter ratio and other limit manufacturing capabilities [1, 10, 11]: femtosecond pulse sequence is proposed to control the free electron density and photon absorption efficiency, control the modification degree of material structure, and increase the etching efficiency by 37 times, as shown in Fig. 6.2 (a); a new super diffraction limit processing method for spatial shaping to control local instantaneous electron dynamics is proposed. The processed nanowires have good stability, are easy to pattern, and the linewidth can reach 1/14 of the wavelength, as shown in Fig. 6.2 (b); through time and space shaping, optimization and adjustment of ionized electron density distribution, the limit of the depth-diameter ratio of microhole processing is increased by 100 times (1000:1, 1.5 μm), and the efficiency is increased by 56 times,

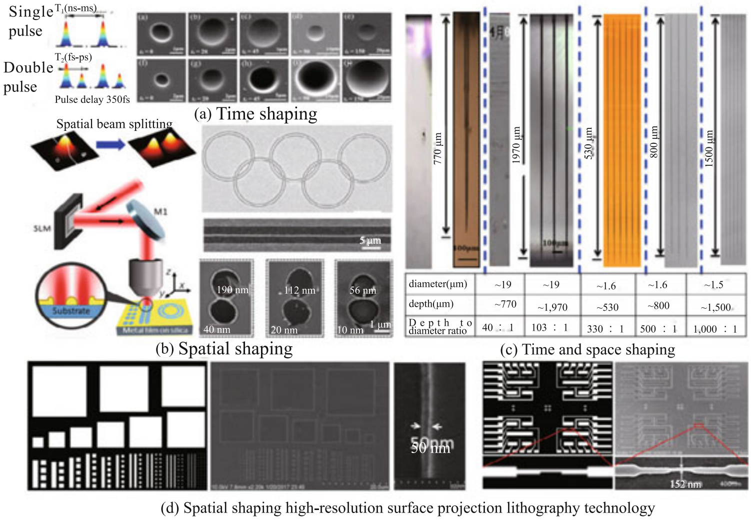

Fig. 6.2 A new method of ultra-fast laser space–time shaping micro-nano machining (**a** Time shaping to improve etching efficiency; **b** Space shape to improve machining accuracy; **c** Space–time shape to improve that depth-diameter ratio limit of micropores; **d** Large-area, high-precision, cross-scale micro-nano process)

as shown in Fig. 6.2 (c); based on the new laser space-time shaping micro-nano machining method, combined with DMD technology, large-area, high-precision, cross-scale micro-nanomanufacturing is realized, and the linewidth resolution is 50 nm, and the linewidth is realized Span 152–140 mm, as shown in Fig. 6.2 (d).

The new method of ultra-fast laser electronic dynamic control processing has attracted extensive international attention, and the related research work has been greatly positively evaluated by more than 70 academicians/scholars (including Nobel Prize winners) from various countries in *Science*, *Nature* and other journals. For example, OSA/SPIE Fellow and Professor P. Herman of the University of Toronto, Canada, cited the new manufacturing method in six places in his specially invited review paper, and listed the proposed new manufacturing method as “the best reported femtosecond laser results (one of the best reported femtosecond laser results)”. Professor Koji Sugioka of OSA/SPIE Institute of Physical Chemistry, Japan, greatly evaluated the new method in nanophotonics, saying that the new method successfully processed 3D micro-nano structures that were “previously inaccessible”. *Laser Focus World* published the topic “Shaping Femtosecond Laser to Change Electronic Dynamics and Improve Ultrafast Laser Processing Quality” to comment on this new method: “Stunning results have been obtained.” The research team was invited to be in *Light: Sci & Appl* of the *Nature* published a 26-page special topic review entitled “New Electronic Dynamic Control Micro-nano Processing Method for Femtosecond Laser Space-time Shaping: Theory, Method, Observation and Application”, summarizing the main scientific research progress of the new method in the past 10 years, and taking it as the only highlight on the front page of the journal website for more than one month. The news platform of the American Association for the Advancement of Science and its journal *Science*, titled "Femtosecond Laser Manufacturing: Realizing Dynamic Control of Electrons", made a special report on the new method, and said that the new method "may bring revolutionary contribution to high-end manufacturing, material processing and chemical reaction control", which was reprinted by more than 10 international mainstream scientific and technological media such as Phys.org.

6.2 Multi-Time Scale Observation of Ultra-Fast Laser Micro-Nanomanufacturing Process

Ultra-fast laser processing is a non-equilibrium, nonlinear and ultra-fast process, which involves many physical and chemical processes, such as laser propagation and material ionization, material phase transition, plasma eruption and radiation, shock wave formation and propagation, and microstructure formation [12]. The characteristic time of the above physical and chemical processes ranges from femtoseconds to milliseconds or even seconds, but they influence each other. Based on this, the research group of Beijing Institute of Technology proposed and established a real-time observation system spanning the multi-time scale of "femtosecond-picosecond-nanosecond-millisecond" (Fig. 6.3), spanning 12 time orders of magnitude, femtosecond laser pump detection, laser-induced breakdown spectroscopy, time-resolved plasma imaging, industrial continuous imaging and other technologies [13] are comprehensively used to capture the process of laser processing of high aspect ratio microholes in real-time by charge-coupled device (CCD) camera on the second time scale; on the nanosecond scale, the eruption process of plasma has been observed by femtosecond laser double pulse induced breakdown spectroscopy system on the picosecond time scale, the generation and evolution of laser-induced plasma/shock wave have been observed by pump-probe microscopy system on the femtosecond time scale, the dynamic propagation process of the femtosecond laser pulse and the ionization process of materials have been observed. Using a multiscale observation system, the regulation mechanism of femtosecond laser spatio-temporal shaping on electron heating, ionization and recombination in manufacturing and its influence on microstructure shaping and formation are revealed. For the first time, panoramic observation (from constant speed to 3.5 trillion times slower) of mass and energy transmission process with electrons as the main energy carrier in the manufacturing process has been realized, providing observation evidence for new manufacturing technologies and promoting the development of ultra-fast technology.

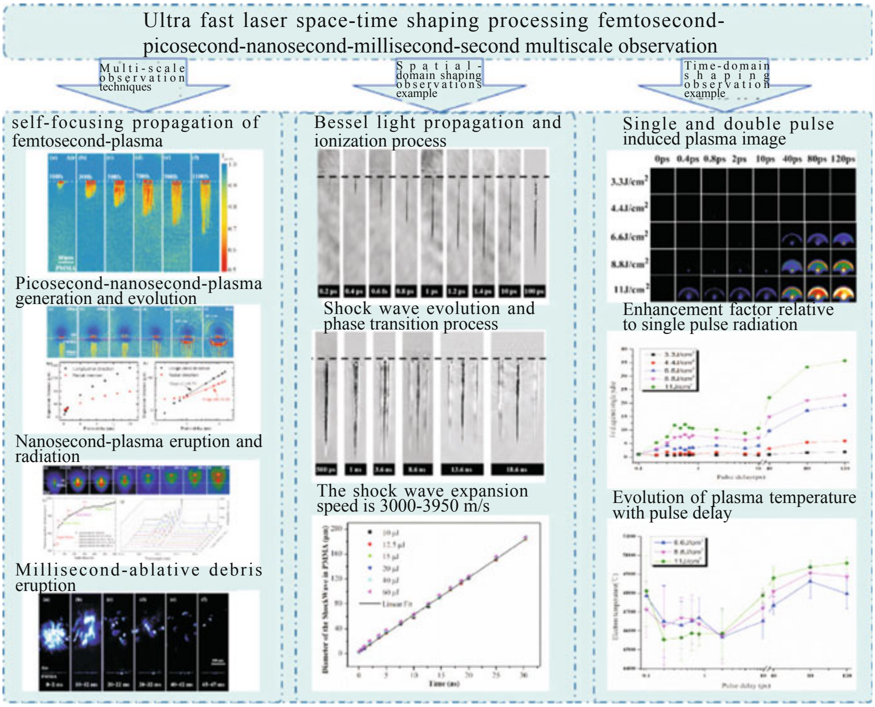

Fig. 6.3 Real-time observation of ultra-fast laser spatio-temporal shaping processing femtosecond-picosecond-nanosecond-millisecond multi-time scale

6.3 Important Engineering Applications of Laser Micro-Nanomanufacturing

Based on the new method of electronic dynamic control processing, laser micro-nanomanufacturing realizes the active control of local instantaneous electronic dynamics in manufacturing for the first time, thereby greatly improving the processing quality, precision, efficiency, depth-to-diameter ratio and other extreme manufacturing capabilities. The application of this new method successfully has solved the technical problems of many important projects and provided strong support for the country's major strategic needs.

6.3.1 Machining and Testing of Target Ball Micro-Hole

As a common structure, micropores are widely used in various fields. However, as the application requirements of major national strategic needs continue to rise, the depth-to-diameter ratio, aperture, shape complexity, and quality requirements of

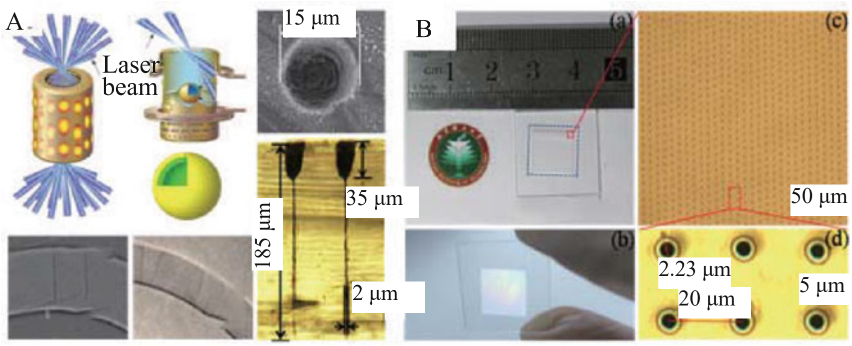

Fig. 6.4 Target ball micro-hole processing for a major national project (**a** Target ball micro-hole processing; **b** Single-pulse ultra-fast laser bessel beam flying drilling method to achieve high-efficiency, high-quality, and high-consistency micro- hole array processing)

micropores are constantly pushing to new extremes. The micro-nanomanufacturing team of Beijing Institute of Technology uses a new method of ultra-fast laser space-time shaping micro-nano processing to address the technical challenges of micro-hole processing in the core structure of a major national project (such as large depth-to-diameter ratio, minimization of recast layers/sputters, hole quality requirements are high; residues in the cavity are minimized; processing efficiency needs to be improved), the spatial distribution of the ultra-fast laser light field is changed through spatial shaping, the instantaneous local electron density distribution and its phase transition process are controlled, and the plasma is optimized and adjusted. The eruption process has overcome the large depth- to-diameter ratio (1000:1, diameter 1.5 μm), high consistency (250,000 holes/cm^2), high quality (no microcracks/recast layer), and high efficiency (100 holes in a single beam/sec), minimization of residues and other problems in the preparation of micropores (Fig. 6.4), and has been selected as the only processing technology for the micropores of the national major projects [14].

In response to the test of the inner and outer diameter and thickness parameters of the target ball in the major national engineering, the Beijing Institute of Technology research team proposed a laser radial polarization differential confocal longitudinal field tomography detection method [15, 16] (Fig. 6.5), using the difference moving confocal technology to improve the axial resolution of the confocal imaging detection technology; using radially polarized tight focus technology and pupil filter technology to significantly compress the focus spot; improving the lateral resolution of the confocal imaging detection system. Reuse image restoration technology further improve the lateral resolving power, and then improve the spatial resolving power of the system. This method, combined with image edge processing technology, can achieve 2 nm axial and 80 nm lateral resolution structural testing, and can be used for nanometer-precision metrological testing and calibration of large-scale and large convex-concave standard samples.

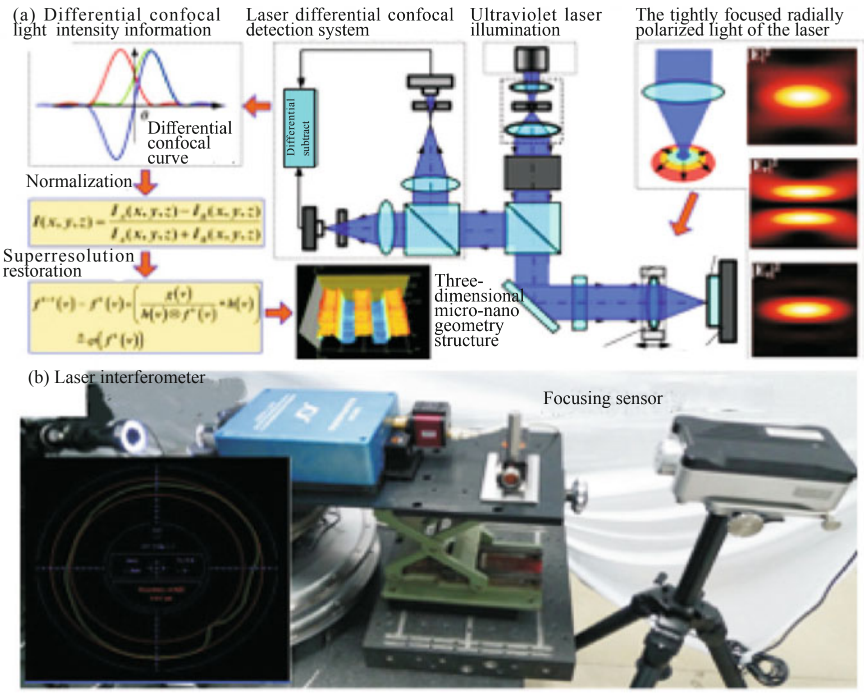

Fig. 6.5 Principle and device of laser radial polarization differential confocal longitudinal field tomography detection method

6.3.2 Manufacturing of New Optical Fiber Sensors

The research team of Beijing Institute of Technology has aimed at the manufacturing problems of 3D micro-nano structures of optical fiber devices and has adopted a new method of femtosecond laser manufacturing to process the invented series of temperature, pressure, vibration and concentration on optical fibers such as pure quartz with high quality and high precision. Sensors [17–19] (Fig. 6.6), as well as signal demodulators and measuring instruments, solve the common bottleneck challenges that severely restrict the development of my country's cutting-edge defense equipment: testing in small areas, high temperature, high pressure, and strong electromagnetic environments. Professor Cusano, editor-in-chief of *Opt Laser Technol*, has commented that the sensitivity of the new sensor is "the highest record currently", and it has been successfully applied to a hypersonic aircraft high temperature strain test, a stealth fighter pressure test, a missile/rocket fuze bridge wire ignition temperature, a gun bore temperature/testing of key physical quantities of major equipment in the fi of defense, such as deformation, provides important support for the development/production of my country's cutting-edge technical equipment.

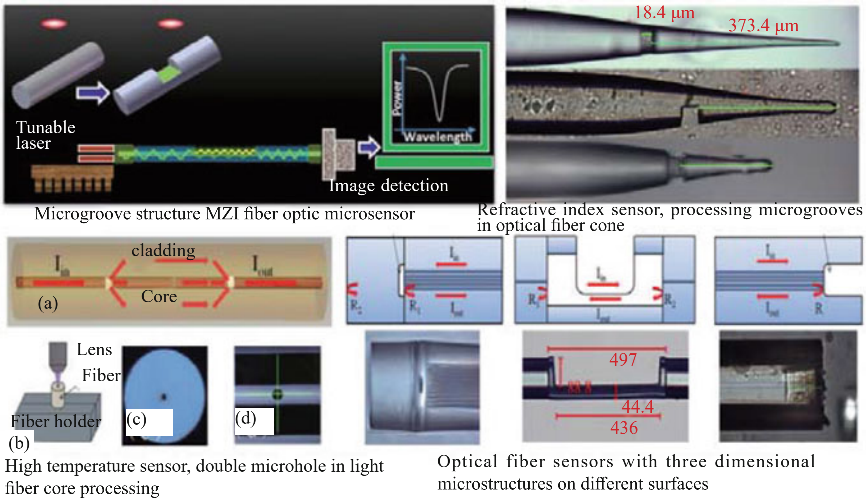

Fig. 6.6 Laser processing series of three-dimensional micro-structure optical fiber micro sensor

Focusing on the two key issues of improving manufacturing accuracy and speed, the research team of Central South University has used femtosecond laser manufacturing technology to achieve nanolevel precision processing of optical fiber micro/nano devices and the manufacturing of new device structures in order to solve the problem of micro-nano structure manufacturing of optical fiber devices. China's independent manufacturing of high-precision, high-performance optical fiber devices provides a theoretical foundation and technical reserves [20, 21].

The research team has clarified the femtosecond laser processing and forming mechanism of the fiber micro/nano structure, and has carried out the analysis of the processing mechanism of transparent media materials. By designing the beam control system and processing system, using bessel to shape the beam, the ablation processing radius in the target quartz glass can be achieved. The adjustable ring structure and micro-channel, realize micro-hole processing with a depth-to- diameter ratio of about 500:1, and realize rapid processing of micro-hole structure and long-period fiber grating on the optical fiber, and the micro-hole structure fiber has high refraction rate sensing characteristics; a high-precision and fast fiber micro-nano structure manufacturing method based on multi-beam simultaneous irradiation and femtosecond laser nonlinear effects is proposed, and the long-period fiber is written by the femtosecond laser point-by-point method and line scanning method respectively. The influence of different parameters (grating period, grating length, duty cycle, scan times, etc.) on the transmission spectrum of the LPFG under the point-by-point method is analyzad. The temperature characteristics of long-period fiber gratings are theoretically analyzed, and temperature control experiments are carried out on the long-period fiber gratings written by line scan. The experimental

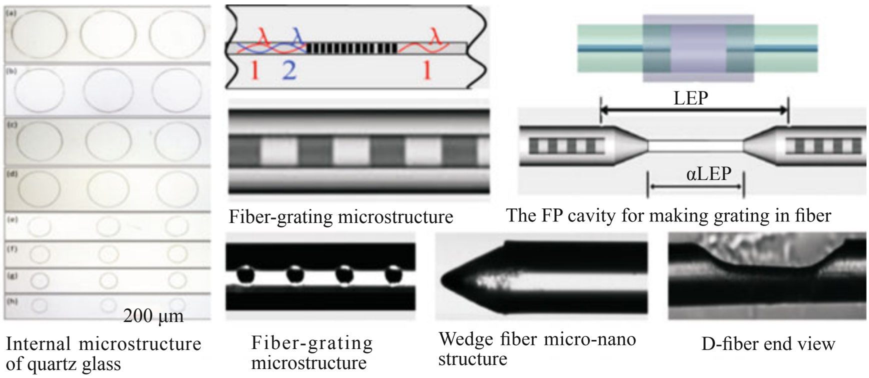

Fig. 6.7 Side view of five-ring micro-nanoscale structure and optical fiber micro-hole

results show that it is suitable for wavelength control in the low temperature range and has high temperature sensitivity in the high temperature range. Part of the result is shown in Fig. 6.7.

6.3.3 Optical Device Manufacturing

Optical device manufacturing has become a difficult problem in the manufacturing field due to its difficult material processing and high precision requirements. For example, infrared guidance window anti-reflection is a key technology for high-speed missiles. The integrated micro-nano structure does not require coating, which not only ensures reliability, but also effectively realizes anti-reflection, which is a key manufacturing bottleneck for the speed increase of high-speed missiles. The anti-reflection of infrared guidance windows has common challenges: difficult to process materials (zinc sulfide, sapphire, diamond, etc.), large area (hundreds of square centimeters), high uniformity, three-dimensional surface micro-nano structure, difficult to pass photolithography, imprinting, and reaction ion etching and other processing. The research team of Central South University and the research team of Jilin University have used ultra-fast laser direct writing to process large-area, highly consistent inverted pyramid-shaped microstructures and hole arrays (period 2 μm, diameter 1 μm, height 1 μm) on the surface of sapphire material; the transmittance reaches 92%~95%; in the incident range of 0~70°, the surface maintains high transmittance [Fig. 6.8 (a)]; at the same time, the new method of ultra-fast laser pulse shaping electronic dynamic control processing is applied , processing large area (φ100 mm) hole array infrared antireflection microstructure (period 3.6 μm, diameter 3.3 μm, depth 0.8 μm) on the surface of zinc sulfide, with a transmittance exceeding 85% [Fig. 6.8(b)] [22].

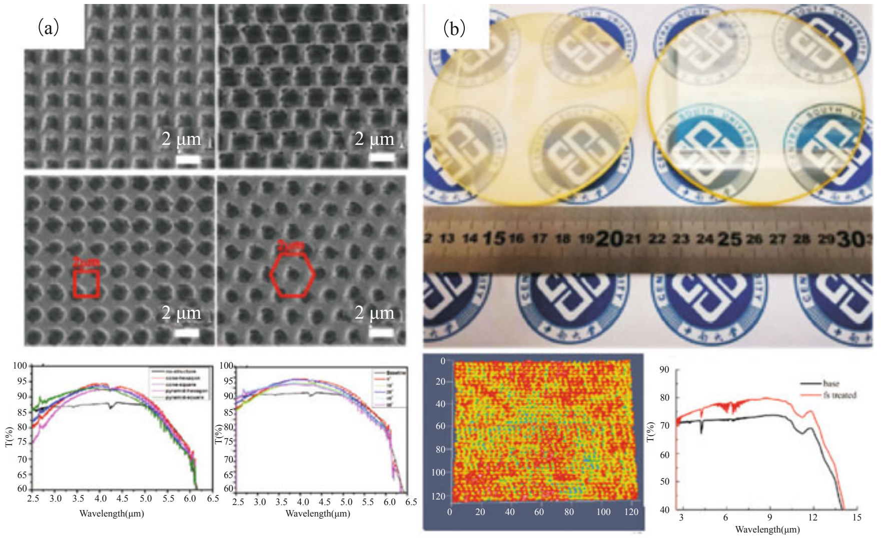

Fig. 6.8 Manufacturing of anti-reflection structures for difficult-to-process materials (**a** Sapphire material; **b** Zinc sulfide material)

The aspheric microlen is also an important optical device, which has important applications in aviation, aerospace and other fields. However, due to its three- dimensional complex and difficult-to-machine curved surface and high precision requirements, it has become a major problem in the manufacture of optical devices. The research team of Jilin University has proposed a new method for processing curved micro-lenses and arrays using femtosecond laser micro-nano processing technology in response to the manufacturing problems of aspherical micro-optical components, laying a theoretical foundation for high-precision, repeatable and high-efficiency laser micro-nano processing with technical support. It provides a new idea for the manufacture of aspheric micro-optical components that are urgently needed in the fields of aviation, aerospace and laser technology. This technology provides novel solutions for basic research and common micro-optics problems faced by frontier fields of national defense applications such as organic electroluminescence, solar cells, high-performance optical fiber sensing, micro- flow and optical flow detection, and filmless anti-reflection infrared guidance [23–26]. Based on the above-mentioned new methods and new technologies, a series of high-performance micro-optical elements represented by shaping microlenses, such as zone plates, aspheric refractive lenses, refractive hybrids, and tunable lenses, have been realized (Fig. 6.9). In addition, in response to the difficult problem of semiconductor diode laser beam shaping, femtosecond laser processing is used to fabricate integrated aspheric microlenses for vertical cavity surface-emitting lasers, which reduces the output laser divergence

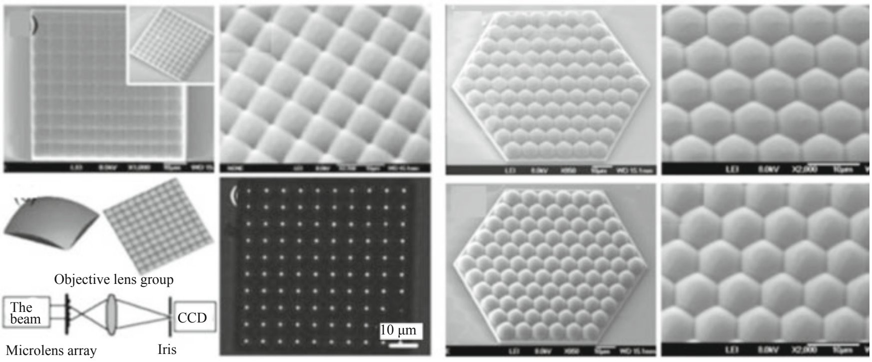

Fig. 6.9 Femtosecond laser processing of high numerical aperture hexagonal close-packed microlens array

angle from 18.16° to 0.86°. For edge- emitting semiconductor diode lasers, asymmetric multi-order zone plates and non- aligned hyperboloid lenses are prepared, achieving ideal shaping effects from 60° on the fast axis and 9° on the slow axis to 6.9 mrad and 32.3 mrad. The efficiency of optical fiber coupling is higher than 80%.

6.3.4 *Preparation of New Materials*

The new method of ultra-fast laser processing not only has an important application in the manufacture of traditional materials, but also shows significant advantages in the preparation of new materials. The Beijing Institute of Technology research team has established a laser-assisted preparation method system for graphene in different dimensions, using the interaction of laser and carbon micro-nano materials to control the local instantaneous properties of the material, and achieve high- precision and high-efficiency manufacturing of multi-dimensional micro-nano functional structures [27–29]: laser direct writing quickly prepares good monodisperse zero-dimensional graphene quantum dots [Fig. 6.10 (a)]; laser micro-nano processing one-dimensional graphene microfibers to obtain walking robots [Fig. 6.10 (b)]; laser rapid radiation direct writing of two-dimensional graphene films to obtain graphene memory diodes [Fig. 6.10 (c)]; laser-assisted deposition to prepare functional three-dimensional graphene foams and other micro-nano functional structures [Fig. 6.10 (d)]. Using the above-mentioned new laser processing methods, a series of functional graphene-based devices have been constructed, which greatly expand the application of new materials such as graphene in the fields of capacitors, infrared sensors, fuel cells, and lithium-ion batteries.

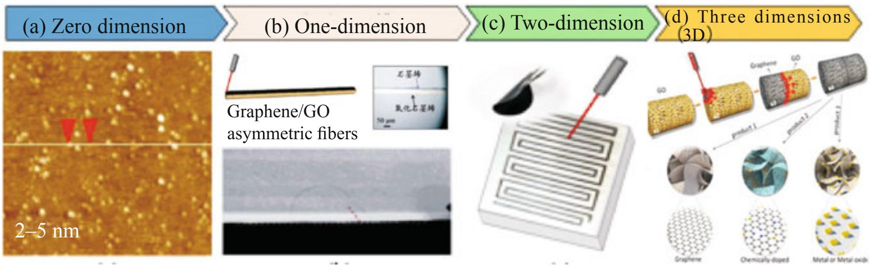

Fig. 6.10 Laser-assisted preparation method of graphene with different dimensions (**a** Zero-dimensional quantum dots; **b** One-dimensional graphene fiber; **c** Two-dimensional graphene film; **d** Three-dimensional graphene foam)

6.3.5 Manufacturing of Nanocrystalline Large-Area Assembly Structure

The nanocrystalline assembly structure provides greater flexibility and possibility for optimizing and regulating the properties of materials, and is of great significance to the development of chemical catalysis, solar energy conversion, and biomedicine. Utilizing the advantages of laser manufacturing, the research team of the Beijing Institute of Technology has established a method of laser-irradiated colloidal nanocrystals to assemble a large area to obtain a micron-scale nano-superstructure. The interaction between the resonant continuous laser and the Plasmon assembly nanostructure is manipulated to control the microstructure of materials the local photothermal activity of the structure, and realizes the *in-situ* welding of the precisely synthesized Plasmonic zero-dimensional nanocrystalline particles to obtain the superstructure. In order to realize the micro-nanomanufacturing and processing of the assembled structure of the zero-dimensional nanocrystalline, the inter-particle Efficient electron transmission and device applications have laid a solid material foundation [30–32]: the precise synthesis of Au@CdS, Au and other Plasmonic zero-dimensional particle assembly films using resonantly coupled continuous laser irradiation can realize the inter-nanoparticle interaction. Nano- welding to form a superstructure, and realize its application in light absorption and flexible devices (Fig. 6.11A); use precise synthesis of doped quantum dot (CdSe: Ag) film, continuous laser-assisted micro cd at 3.1 eV. The test and the transient spectrum test of the femtosecond (95 fs pulse, ~800 nm) laser realized the photo- induced magnetism in the non-magnetic Ag-doped CdSe quantum dots (Fig. 6.11B). Using the above-mentioned new laser irradiation processing method, the nano-particles are processed for micro-nano processing, which greatly expands the bottom-up assembly methodology of zero-dimensional plasmonic nanocrystals and doped quantum dots, and realizes its use in photoelectric detection, photomagnetic New applications in the fields of coupling and spintronics.

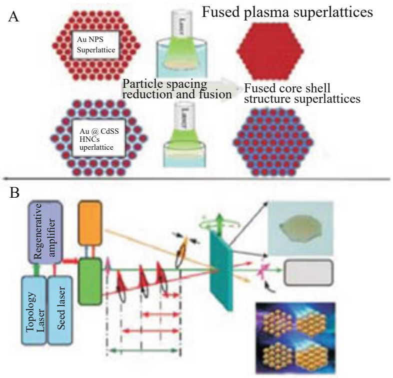

Fig. 6.11 **A** Use 532 nm (6.68–13.37 W·cm^{-2}) continuous laser irradiation to accurately synthesize plasmonic zero-dimensional particle assembly films such as Au @ CdS, Au, etc., to realize nano-welding between nanoparticles into a superstructure, and realize its application in light absorption (a) and flexible devices (b); **B** Use the precise synthesis of doped quantum dots (CdSe: Ag) film, continuous laser-assisted MCD test at 3.1 eV and femtosecond (95 fs pulse, ~800 nm) laser transient spectroscopy test, realized the light in non-magnetic Ag- doped CdSe quantum dots induced magnetism (a–e)

6.4 Laser Micro-Nanomanufacturing Equipment

The high-efficiency processing of large-area micro-nano 3D topography is a major international problem. The core technical problem is how to efficiently transform the massive design data into 3D micro-nano topography. Based on a new method of laser spatial shaping, the research team of Soochow University has proposed a lithography technology based on phase-space light hybrid modulation and digital light field, using "micro-nano structure light field" frame-by-frame digital rolling stack (integration) technology (12,000 frames)/sec, 1920×1080 data/frame, 3D navigation flight exposure mode, has overcome the major problems of efficient processing of complex micro-nano 3D topography on warped surfaces, and has successfully developed the "Large-area micro-nano 3D direct writing equipment MiScanV" (Fig. 6.12), filling the industry gap, and has successfully applied to large-area flexible functional materials, large-size capacitive sensors, new display devices, MEMS devices and other high-tech research fields for national defense and civil use. In the National "Twelfth Five-Year" Science and Technology Innovation Achievement Exhibition, large-area micro-nano 3D direct writing equipment has been exhibited as a landmark achievement in the advanced manufacturing field of the national "863 Program". At present, the equipment has been used in the development and manufacturing of micro-nano materials and devices in dozens of units such as China Electronics Technology Group Co., Ltd., Tsinghua University, Hong Kong Polytechnic University, and has been exported to Russia, Israel and other countries.

In response to the lack of technical means of "sub-nanometer precision control" in the development of photonic chips, 3D displays, and optical waveguide devices,

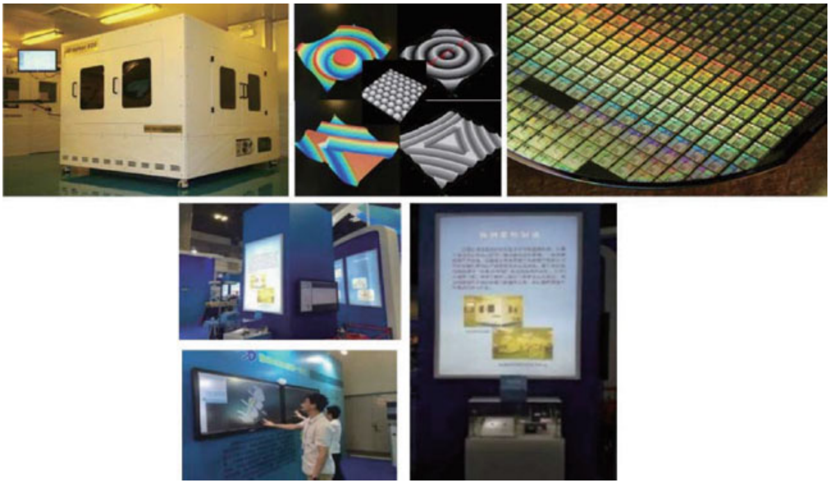

Fig. 6.12 Large-area micro-nano 3D direct writing device MiScanV and its iconic results

the research team of Soochow University has invented a new method of nanolithography with "five-dimensional micro-nano optical field control" (structural unit). The structure resolution is close to λ/4, which is 2 times higher than that of the projection lithography system; and has successfully developed a nanolithography equipment "NanoCryatal" with a structure modulation accuracy of <1 nm. The field size is 100–250 μm, the structure size in the light field is >90 nm, the format is 6–32 inches, the structured light field lithography mode, the rate is 100–500 mm^2/min, which fills the gap in the industry and realizes the industrial application (Shanghai Jiaotong University, The Hong Kong Polytechnic University and many companies).

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