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MODERN GaAs MATERIALS

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The main current modernized methods for growing GaAs single crystals, which are based on the well-known and widely used Czochralski methods, directional crystallization in Bridgman variants and crystallization in a moving gradient temperature, or a combination of these methods are analyzed. The work studies the influence of crystal growing methods on the properties of semiconducting and semiinsulating gallium arsenide for various applications. The results of obtaining GaAs single crystals of various diameters obtained by world competitive manufacturers are systematized. The degree of perfection of the grown GaAs single crystals plays a key role in obtaining the necessary critical properties of GaAs for microelectronics today.

INTRODUCTION

Gallium arsenide is widely used worldwide in various industries, including wireless communications, optoelectronics and solar cells, microwave integrated circuits (MICs), monolithic microwave integrated circuits (MMICs), discrete microwave (MHF) transistors, light-emitting diodes (LEDs), laser diodes, photodetectors and nuclear radiation detectors [1–4].

The main advantages of GaAs are: high electron mobility, low resistivity, wide band gap, low noise, thermal stability. GaAs has higher electron mobility compared to Si, which provides faster device operation and higher frequency capabilities [5]. This property is crucial in applications requiring high-speed signal processing, such as wireless communication systems and high-speed data transmission. In turn, the low resistivity of GaAs provides efficient charge transport and allows devices to operate at higher speeds. The wide band gap of GaAs allows for the creation of high-performance optoelectronic devices: LEDs, laser diodes and photodetectors, and enables the use of GaAs in optical communication, in display technologies. In addition, the wide band gap provides emission and detection of light in a wider range of wavelengths, which leads to increased device performance and efficiency. The low noise characteristics of GaAs make it suitable for applications where high signal-to-noise ratios are required. This is important when using this material in telecommunications and radio frequency systems. GaAs has excellent thermal stability, allowing devices based on it to withstand high operating temperatures without significant degradation of performance. It should be noted that the most promising applications of the semiconductor GaAs for electronic devices require the use of high-purity single-crystal materials. The synthesis and purification of bulk polycrystalline semiconductor material is the first step in the fabrication of an electronic device. Then the synthesized material is melted and regrown using a strictly controlled process to form single-crystal ingots that can be processed into wafers.

GaAs GROWTH METHODS

The following growth methods are used in the production of GaAs single crystals: Czochralski method with liquid sealing of the melt with a layer of boric anhydride (Liquid Encapsulate Czochralski – LEC), methods of directed vertical and horizontal crystallization in Bridgman variants (Horizontal Bridgman – HB, Vertical Bridgman – VB), or the moving temperature gradient crystallization method (Horizontal Gradient Freeze – HGF, Vertical Gradient Freeze – VGF) [6–8]. In some cases, the Bridgman-Stockbarger method is used [9]. The main difference between the Bridgman-Stockbarger method and the basic Bridgman method is the improved temperature control system, which is achieved by using two temperature zones instead of one. In the basic Bridgman method, the ampoule with the melt moves through a single zone with a temperature gradient, while the Stockbarger modification uses two zones – hot and cold, with an insulating partition between them – for more precise control of the crystallization process and obtaining more homogeneous crystals. The properties of single crystals grown by these methods differ, which allows you to choose a process that will allow you to produce wafers that best meet technical needs.

BRIDGMAN METHOD

Bridgman crystal growth is based on the directional crystallization of a melt. This is achieved by transferring the melt from a high-temperature zone of the setup (above the melting point of GaAs) to a low-temperature zone (below the melting point of GaAs) (Fig. 1). The presence of a seed ensures that the single crystal grows along a specific crystallographic orientation. In the Bridgman method, only the relative movement between the crucible and the heater system is important. VB or HB growth systems usually consist of a single- or multi-zone furnace. A single-zone furnace has a parabolic temperature profile along the entire length of the furnace, with the highest temperature in the center. On both sides of the hot zone there is a temperature gradient that is used during crystal growth. For a multi-zone furnace, specific temperature gradients can be set between the different zones.

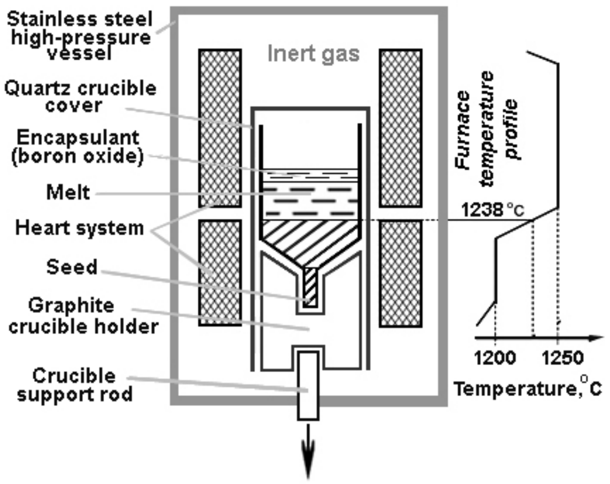

Fig. 1. Principle scheme of a modern arrangement for growing GaAs single crystals in an open crucible using the vertical Bridgman method

Crucible surface preparation is critical for all GaAs growth methods. Pyrolytic boron nitride crucibles require high-temperature annealing in air to form a thin surface layer of boron oxide. Quartz crucibles require chemical etching in hydrofluoric acid. When growing GaAs crystals in containers, melt encapsulation serves a dual function: first, it prevents the escape of volatiles from the melt, and second, it prevents contact between the gallium arsenide melt and the crucible walls during growth, preventing the formation of secondary nuclei that lead to the formation of polycrystalline grains. In the VB growth method, polycrystalline gallium arsenide, a single-crystal GaAs seed, and a molten encapsulating material (B_2O_3) are placed inside the crucible. In the sealed crucible configuration, the crucible should be placed in a separate quartz container and sealed under a vacuum of 10^{-6} Torr. If vapor pressure control is required, for example with As or P, a separate reservoir containing the appropriate element is also inserted into the vacuum quartz container. For experiments performed in the open crucible configuration, the growth chamber should first be evacuated to a high vacuum ($\sim 10^{-6}$ Torr) and then filled with an inert gas to create a high pressure, as in LEC systems [10]. After the crucible is placed in the growth chamber and the inert gas pressure is stabilized, the furnace temperature is raised to a temperature above the melting point of gallium arsenide. Then the crucible is slowly moved to the hot zone. Crystal growth is initiated by cooling the melt from the seed end. This is done by moving the crucible to a colder section of the furnace. As the crucible moves, the temperature at the bottom of the crucible drops below the solidification temperature (the melting point of GaAs), and the melt begins to crystallize at the interface between the seed and the melt. After the melt has completely cooled below the melting point of the material, the entire molten charge is converted into a single-crystal solid ingot. After the entire molten charge has solidified in a direction, the furnace is cooled to room temperature at a rate of 25…50 °C/h [10]. The cooling rate after growth must be controlled to reduce stresses resulting from differential expansion and contraction of the crystal, the encapsulating material, and the crucible walls. The crystal and seed can be removed from the crucible by dissolving the encapsulating material.

The HB and VB processes are similar. However, there are certain advantages and disadvantages to the two methods. The wafers from crystals grown by the VB method have a perfectly round shape, in contrast to the D-shaped wafers of crystals grown by the HB method. Round wafers are desirable for large-scale production. The main advantages of crystals grown by HB are the high crystalline quality of the crystals (low dislocation density) and the control of stoichiometry along the entire length of the crystal. In the HB method, the crystal experiences less stress due to the free surface at the top of the melt. During growth, the solid does not touch the crucible from above and therefore expands freely.

A more comprehensive study of the Bridgman method was conducted by University Wafer, Inc. They proposed a modified Bridgman method. This new method eliminates deformation and allows for the production of a crystal without twins, and also allows for better control of the temperature gradient at the melt/crystal interface when the crystals are just forming and are undergoing a growth phase that involves the addition of new atoms, ions, and polymer compounds. This method can be used to grow large single crystals of GaAs. The initial material for this process has a strict stoichiometric composition that limits the evaporation of the melts. The temperature gradient is 20…40 °C/cm at the solid-liquid interface, and the optical uniformity is high. This modification of the Bridgman method is promising for the serial production of GaAs semiconductor crystals.

LEC METHOD

The LEC equipment consists of a graphite heating system surrounded by thermal insulation and a water-cooled pressure vessel with sealed passages for movement and rotation of the crystal and crucible, respectively (Fig. 2). The crucible, made of pyrolytic boron nitride contains a GaAs melt covered with liquid boron oxide encapsulated to prevent arsenic loss during melt growth. LEC technology can combine the process of GaAs synthesis (at a pressure of 60…70 atm) and single crystal growth under pressure (20…30 atm). To ensure the required electrophysical parameters, gallium and arsenic with a purity of at least 6N–7N and crucibles made of pyrolytic boron nitride are used. A feature of the method is that the growth is carried out at rather large axial and radial temperature gradients near the crystallization front, resulting in a high dislocation density (ND – from $1 \cdot 10^4$ to $2 \cdot 10^5$ cm^{-2}) [11].

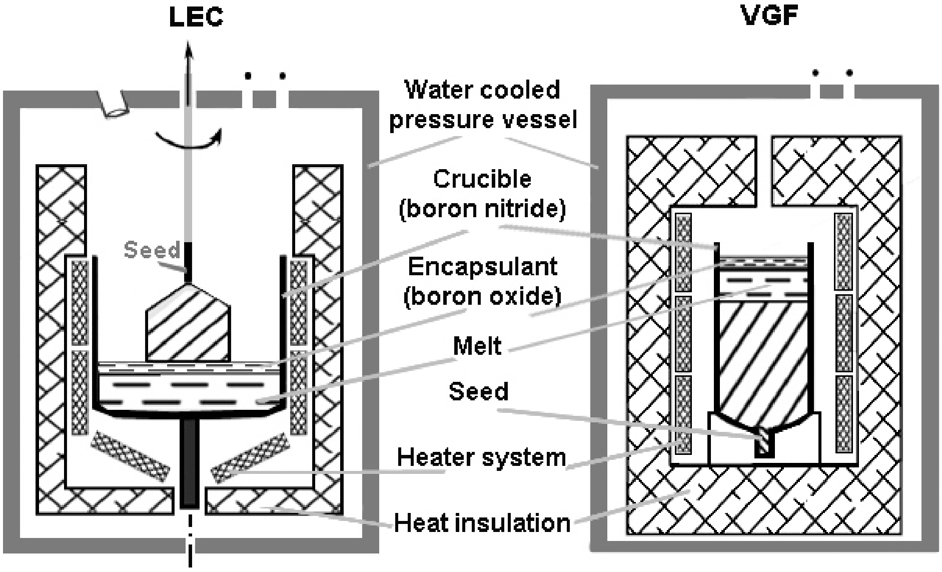

Fig. 2. Principle schemes of a modern arrangements for growing GaAs single crystals using the LEC and VGF methods

In [12], the growth of GaAs single crystals by the Czochralski liquid encapsulation (LEC) method was considered. GaAs ingots grown by this method had a diameter of 2…2.5 inches and a weight of 1…1.5 kg. Semi-insulating crystals of n-type, p-type and doped, as well as undoped semi-insulating crystals were grown using <111> and <100> seed crystals.

The development of the method is towards increasing the loading and increasing the diameter of the crystal being grown. With this method, crystals with a diameter of up to 150 mm are grown. Systems with multiple heaters allow for proper adjustment of the solid-liquid interface, as well as control of the temperature of the crystal emerging from the boron oxide to avoid selective evaporation of arsenic and reduce residual deformation. The extractor can be equipped with a gas supply system to control the C and O potentials, as well as a fully computerized process and diameter control system. Growth is usually carried out using a seed and crucible rotating in opposite directions, with a growth rate of 5 to 10 mm/h and a seed orientation of <100> in an atmosphere containing nitrogen and CO at a pressure of more than 0.2 MPa.

VGF METHOD

The VGF method of growth is one of the most common processes for creating GaAs wafers. The vertical gradient freezing method allows for the production of crystals with a reduced density of dislocations – this is a type of crystal growth process that involves the simultaneous formation of a crystal and a melt (see Fig. 2). This process is characterized by the spatial extents of the crystal and the melt varying in time and involves the solution of two related problems with free boundaries. The solution process is usually obtained by the finite element method, which converts the simplified spatially distributed representation of the system into a spatially concentrated representation. Freiberger Compound Materials offers the VGF method, in which a pyrolytic crucible made of boron nitride is loaded with pre-synthesized GaAs and boron oxide. Then the filled crucible is placed in a furnace assembled from a series of separately controlled zones that allow the creation of two zones of close to constant temperature above and below the melting temperature, and a controlled (small) temperature gradient between them. Crystal growth is initiated by a predominantly <100> oriented seed fixed at the bottom of the crucible and continues with the solid-liquid interface moving upwards. The temperature field is shifted by controlling the heater system, which provides a given growth rate and interface shape. The low axial temperature gradient, typically below 5 K/cm, results in crystals with a very low dislocation density.

In this work, the parameters and factors affecting the growth of VGF-GaAs are optimized using computer modeling [13].

COMPARISON OF METHODS

The key difference between the methods is that the Bridgman and VGF methods are directional crystallization methods, where the crucible or furnace moves relative to each other to create a crystallization front, while the Czochralski method is a method of pulling a crystal from a melt, where a seed pulls a single crystal and moves upward, while the melt remains stationary. The properties of single crystals grown by these methods differ, which allows you to choose a process that will allow you to produce wafers that best meet technical needs.

Device performance and degradation mechanisms are critically affected by bulk properties. Consequently, significant efforts have been made to reduce crystal defects and improve the uniformity of physical properties, which is associated with the need to increase the crystal diameter. This goal is not easily achieved due to the proportionality between crystal diameter and dislocation density. Reducing the dislocation density (below 10^4 cm^{-3}) by reducing the nonlinearity of the thermal field during LEC growth has led to the development of fully encapsulated and vapor pressure-controlled Czochralski methods (FEC and VCZ, respectively). Another direction to achieve the same goal has been the improvement of the vertical gradient Bridgman (VB) and vertical gradient freezing (VGF) methods. In [14], the current state, advantages and disadvantages, as well as future developments of the growth methods are summarized (Table 1). Some fundamental problems of heat transfer, dislocation dynamics (polygonization), and growth phenomena associated with non-stoichiometry are discussed in detail.

Table 1

Comparison of industrially applied methods for bulk GaAs growth [14]

Growth methods	LEC	VCZ	HB	VB/VGF
Capability for low dislocation density	Poor	Good	Very good	Very good
Uniformity of dislocation distribution	Moderate	Good	Good	Good
Cell,cellsize	Small	Large	Very large	Very large
C-Control	Very good	Not solved	Poor	Not solved
Stoichiometry/EL2 control	Good	Good	Poor(?)	Very good
Crystal length	Very good	Limited	Good	Good
Crystal diameter	Very good	Very good	Limited	Very good
Background impurities	Low	Low	Low	Low
Technical realization	Good	Possible	Good	Good
Commercial features				
Investment cost	High	Very high	Low	Low
Operational cost	High	High	–	Low
Process maturity	Very high	Low	High	High
Productivity	High	Medium	Medium	Low
Fields of application	μe-devices/ implantation	μe-devices/ implantation, LEDs	LEDs, LDs	μe -devices/expitaxy, LED, LDs

The vertical gradient freezing (VGF) method allows to obtain a material that is well suited for devices with high current density (HBT, LEDs, lasers, etc.), while the Czochralski liquid encapsulation (LEC) method is best suited for devices with a large area where the current density is not so high (MESFET, pHEMT, etc.). The VGF method allows to obtain crystals with a low dislocation density. The Bridgman method, in turn, is a reliable method for obtaining single-crystal ingots, but does not provide uniform properties throughout the grown crystal.

At present, there are several types of GaAs single crystals. Semi-insulating (SI) GaAs with high resistivity (10^{-7} Ω·cm) is used in the production of high-frequency integrated circuits and discrete microelectronic devices. In addition to high resistivity, (SI) GaAs single crystals must have high carrier mobility values and high macro- and microscopic uniformity of the distribution of properties, both in the cross section and along the length of the grown ingots. There are silicon-doped semiconducting (SC) GaAs of n-type conductivity with a low dislocation density. Single crystals of heavily doped silicon ($10^{17}…10^{18}$ cm^{-3}) GaAs, in addition to high conductivity, must have a fairly perfect crystal structure. Single crystals GaAs with $N_D < 10^4$ cm^{-2} are considered suitable for the production of LEDs, and for lasers – with $N_D < 2·10^3$ cm^{-2}. The semiconducting GaAs is used in optoelectronics for the manufacture of injection lasers, light-emitting and photodiodes, photocathodes, serves as a material for microwave oscillation generators, and is used for the manufacture of tunnel diodes. Single crystals of gallium arsenide doped with chromium are used in infrared optics. In addition, single crystals of GaAs doped with zinc or tellurium are used in the manufacture of optoelectronic devices.

GaAs MANUFACTURING COMPANIES

Currently, the main global companies involved in the production and sale of gallium arsenide-based materials are: Freiberger Compound Materials, AXT, Sumitomo Electric, China Crystal Technologies, CMK (The Gallium Arsenide Company, Slovakia), Shenzhou Crystal Technology, Tianjin Jingming Electronic Materials, Wafer Technology, and others. It should be noted that the leading global manufacturers use growth facilities of their own design.

At present, three methods are used to grow GaAs single crystals, but the most widely used mass production method is the VGF method. Most of the world's manufacturers of single crystals and GaAs wafers use this method. It allows you to obtain not only heavily doped material, but also undoped semi-insulating GaAs. Unlike crystals obtained by the LEC method, single crystals obtained by the VGF method have a higher level of perfection of the crystal structure. This technology is not characterized by the strong dependence of the dislocation density level on the ingot diameter inherent in the LEC method and the higher mechanical strength of single crystals. Equipment for growing using the VGF method is five to seven times cheaper than the installations used for the LEC method. In both technologies, the main task is to increase the productivity of the growth equipment, increase the length of the ingots grown and their diameter. There are multi-crucible growth installations, when six or more crucibles are located under the hood of one installation, each in its own container with an independently regulated temperature. Currently, this technology produces ingots of “optoelectronic” quality with a diameter of 2 to 8 inches.

Freiberger Compound Materials was formed on the basis of the State Institute of Non-Ferrous Metals, founded in 1949. It was created to develop and produce ultrapure and semiconductor materials, such as Ge, Si, Ga, As and others. Particular attention was paid to the development of technologies for the production of materials necessary for the electrical industry. The materials were initially produced on pilot lines in the laboratories of the institute. Table 2 shows Freiberger's products based on GaAs single crystals grown by various methods.

Table 2

GaAs products Freiberger (Germany)

Diameter, mm	Conduction range	Dopand	Etch Pit Density	Growth method	Application
200	SI (n-type)	UN/C	≤12,000	VGF	HBT, pHEMT, Solar
150	SI (n-type)	UN/C	≤10,000	VGF	HBT, pHEMT, Solar
150	SC (n-type)	Si	≤3,000	VGF	LED, HBLED, Solar
150	SC (n-type)	Si	≤50	VGF	LASER
150	SC (p-type)	Zn	≤5,000	VGF	LED, HBLED, Solar
100	SI (n-type)	UN/C	≤7,500	VGF	HBT, pHEMT
100	SI (n-type)	UN/C	≤100,000	LEC	pHEMT, MESFET
100	SC (n-type)	Si	≤3,000	VGF	LED, HBLED, Detector
100	SC (n-type)	Si	≤100	VGF	LASER
100	SC (n-type)	Te	≤100,000	LEC	LED
100	SC (p-type)	Zn	≤3,000	VGF	LED, HBLED, Detector, Solar

For 30 years, Sumitomo Electric (Japan) has maintained its leading position as the world's largest producer of gallium arsenide (GaAs) and indium phosphide (InP). One of Sumitomo's key strengths as a supplier of advanced semiconductor materials is its diversified portfolio of crystal growth technologies for both epitaxial and bulk crystal growth. Sumitomo Electric produces 6-inch diameter semi-insulating GaAs wafers using the VB method with an average etch pit density (EPD) ranging from 3 to $6\cdot10^3$ dislocations/cm². This is a significant improvement over wafers grown using the LEC method (around $1.0\cdot10^4$ dislocations/cm² and above). GaAs-based products Sumitomo Electric Industries (Japan) are presented on Table 3.

Table 3

GaAs-based products Sumitomo Electric Industries (SEI), Japan

Application	GaAs Sub Semi-Insulating Application	LED		LASER		
Conduction Type	Semi-Insulating	n-Type		n-Type		
Growth Method Dopant	VB·C-Controlled	VB·Si		VB·Si		
Carbon concentration, cm^{-3}	Controlled ($0.5 \sim 20\cdot10^{15}$)	–		–		
Carrier concentration, cm^{-3}	$\le 1\cdot10^8$	$2 \sim 35\cdot10^{17}$		$0.7 \sim 4\cdot10^{18}$		
Resistivity, Ω·cm	$\ge 8\cdot10^7$	$1 \sim 15\cdot10^{-3}$		$1 \sim 6\cdot10^{-3}$		
Mobility, $cm^2/(V\cdot s)$	≥ 3000	$1.4 \sim 3.5\cdot10^3$		$1.2 \sim 3\cdot10^3$		
EPD average, cm^{-2}	≤ 10000	$\le 5\cdot10^3$		$\le 1\cdot10^2$	$\le 5\cdot10^2$	
Diameter, mm	150	100	150	76	100	150

Table 4

Characteristics of single-crystal GaAs offered by CMK

	GaAs semi-insulating, undoped	GaAs semiconducting, p-type and n-type
Diameter	wafers: from 2 "up to 4" ingots and synthesis: from 2 "up to 6"	
Thickness	wafers: from 325 up to 750 um ingots and synthesis: from 2 "up to 6"	
Dopant	–	Zinc, Silicon, Tellurium
Carrier concentration	–	n-type $1 \cdot 10^{16} \ldots 2 \cdot 10^{18}$ cm^{-3} p-type $1 \cdot 10^{16} \ldots 5 \cdot 10^{19}$ cm^{-3}
Crystal orientation	(100), (110), (111)	
Resistivity	$>1 \cdot 10^{7}$ Ω·cm	$>1 \cdot 10^{-3}$ Ω·cm
Hall mobility	>6000 cm$^{2} \cdot$V$^{-1} \cdot$s^{-1}	n-type $1 \cdot 10^{16} \ldots 2 \cdot 10^{18}$ cm^{-3} p-type $1 \cdot 10^{16} \ldots 5 \cdot 10^{19}$ cm^{-3}
Etch pit density (EPD)	$<1 \cdot 10^{4}$ cm^{-2}	LEC: $<7 \cdot 10^{4}$ cm^{-2} vertical directional crystallization method: $<5 \cdot 10^{3}$ cm^{-2}

Sumitomo has focused on optimizing the VB growth process to achieve lower dislocation density, reduced residual strain, and improved wafer uniformity for improved device performance.

CMK (The Gallium Arsenide Company, Slovakia) produces semi-insulating and semiconductor gallium arsenide wafers and ingots obtained by the LEC method or vertical directional crystallization method. The required electrical parameters are achieved by using 6N purity raw materials (gallium and arsenic). To obtain the required properties of doped GaAs, dopants are used: zinc, silicon and tellurium. The characteristics of single-crystal GaAs offered by CMK are given in Table 4. Production includes the growth of single-crystal and polycrystalline gallium arsenide in crucibles made of pyrolytic boron nitride and ultra-pure quartz. The company produces single-crystal wafers and solid ingots with a diameter of 2…4 inches, suitable for epitaxy and use in high-frequency and optoelectronic devices.

CONCLUSIONS

The main methods for growing GaAs single crystals today are the Czochralski, Bridgman and Vertical Gradient Freeze (VGF) crystallization methods. Depending on the type of grown crystal, different ways of using GaAs materials are possible: for creating microwave integrated circuits, discrete ultrahigh-frequency transistors, LEDs, laser diodes, nuclear radiation detectors, in wireless communications, optoelectronics and solar cells. The VGF method has become the most widespread. This method allows you to obtain not only heavily doped material, but also undoped semi-insulating GaAs. Unlike crystals obtained by the LEC method, single crystals obtained by the VGF method have a higher level of perfection of the crystal structure, are not characterized by a strong dependence of the dislocation density on the ingot diameter, inherent in the LEC method, and higher mechanical strength of single crystals. Equipment for growing by the VGF method is much cheaper than the installations used for the LEC method. In all technologies, the main task is to increase the productivity of the growing equipment, increase the length of the grown ingots and their diameter. As the technologies for obtaining pure GaAs wafers develop and improve, gallium arsenide-based materials will play a crucial role in shaping the future of semiconductor technologies.

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СУЧАСНІ GaAs-МАТЕРІАЛИ

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Зроблено аналіз основних сучасних модернізованих методів вирощування монокристалів GaAs, які базуються на добре відомих широко використовуваних методах Чохральського: спрямованої кристалізації у варіантах за Бріджменом та кристалізації в градієнті температури, що рухається, або комбінації цих методів. У роботі вивчається вплив способів вирощування кристалів на властивості напівпровідникового та напівізолюючого арсеніду галію для різних застосувань. Зроблено систематизацію результатів щодо отримання монокристалів GaAs різного діаметра, отриманих світовими конкурентоспроможними виробниками. Ступінь досконалості монокристалів GaAs, які вирощуються, відіграє ключову роль для отримання необхідних критично важливих властивостей GaAs для мікроелектроніки на сьогоднішній день.