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Silicon Crystal Growth and Processing Technology: A Review

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ABSTRACT: Crystal growing and shaping technologies are being influenced by a combination of new discoveries in materials properties and new requirements from the marketplace. Key elements of change in crystal growth include control of carbon and oxygen, coupled with a transition to computerized large-charge growers, and investigation of magnetic field influence.

Materials shaping is being driven by stringent quality requirements coupled with newly available processing technologies including laser marking, gettering, and edge profiling. New instruments for inspection of particles, material composition, and wafer flatness are contributing to an overall trend towards automation of critical processes. Planarity of the polished slice continues to be a significant concern, since some complex lithography processes show performance gains with ultraflat wafers.

KEY WORDS: silicon, crystal growth, materials processing, shaping, laser marking, inspection instruments, wafer quality

Crystal Growing

Virtually all semiconductor-device technology employs a crystalline material as its starting point. The term *crystal* refers to a material that is highly ordered on a long-range atomic basis. The process of converting a random-ordered or polycrystalline material into one of long-range order is known as crystal growing.

The physics of semiconductor-crystal growth generally is based on establishing thermal and geometrical conditions such that a liquid is solidified in a controlled interface. Most electronic crystal production is currently centered on silicon and utilizes the technique invented by Czochralski (CZ)² and developed by Teal and Little³.

In the CZ process, crystal growth begins with the melting of a charge of

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² Gatos, H. C. in *Crystal Growth: A Tutorial Approach*, W. Bardsley et al., Eds., Proceedings of Third International Summer School on Crystal Growth—1977, North-Holland, The Netherlands, 1979.

³ Teal, G. K., *IEEE Transactions*, Vol. ED-23, No. 7, July 1976, pp. 621-39.

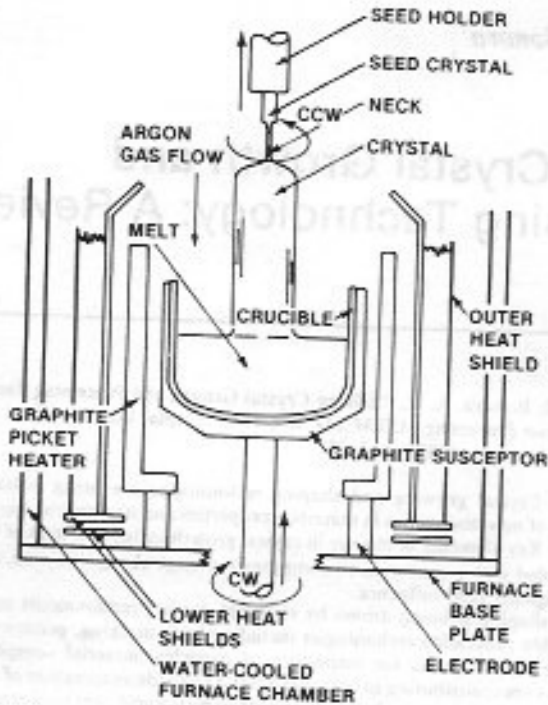


FIG. 1—Schematic cross section of hot zone in a typical furnace for growing single-crystal silicon by the Czochralski process, showing relationships among melt, crystal, heater, and heat shields.

polycrystalline material in a suitable crucible, using surrounding heater(s) and heat shields to establish carefully controlled axial and radial temperature gradients in the melt. A typical furnace for implementing this process is shown schematically in Fig. 1. A seed crystal with the desired crystallographic orientation is dipped into the melt at a temperature near the solidification point. If conditions are correct, it will be possible to begin raising the seed crystal in such a manner that the material makes the transition to a solid-phase crystal at the solid-liquid interface. The newly solidified material replicates the crystalline orientation of the seed. By regulating the temperature and seed-pulling rate, the diameter of the growing crystal can be controlled.

Growth Operations

A prepared polycrystalline charge is loaded into a clean furnace, after which the assembly is checked for leaks and filled with an inert gas, usually argon. With applied power ranging from 50 to 100 kVA, meltdown of the charge proceeds. When the melt reaches "dip-in" temperature (approximately 1420°C), the seed is dipped, and the necking and shouldering procedures follow. Operator skill remains a crucial factor throughout these opera-

tions. Once the crystal reaches the diameter desired, the automatic-diameter-control (ADC) system is turned on and closed-loop controls regulate the diameter.

The process continues through body growth. As the crystal is pulled, the melt level in the crucible drops relative to other hot-zone components. Because this changing melt level creates undesirable changes in thermal profiles at the growth interface, an electromechanical crucible-lift system is normally provided to elevate the crucible continuously during the growth process. The rate of lift, in turn, affects other growth parameters, so the furnace operator must monitor visual indications of structure on the growing crystal to assure that crystal pull speed and crucible lift rate are appropriate.

Body growth proceeds at pull speeds ranging from 50 to 100 mm/h, until only a small volume of melt remains in the crucible; at this point the round-off procedure begins. Structure must be maintained meticulously during round-off, because crystal defects such as "slip" can propagate up through the still-plastic crystal body. After round-off, power to the furnace is turned off and the crystal is allowed to cool. The crystal may later be heat treated to stabilize electrically active oxygen.

Crystal Evaluation

The completed crystal is immersed in an etchant designed to highlight defects such as slip. When the shoulder and round-off portions have been cropped with a diamond saw, resistivity and type measurements are made and recorded for the crystal ends. The cropped ingot is ground to the desired diameter, and may be rechecked for axial profile by taking resistivity readings along the length of its cylindrical surface.

At a location determined by atomic orientation, a flat is ground along the length of the ingot; this will serve as a reference plane for later operations. The crystal may now be sliced into wafers or cropped into segments for specific end users.

CZ Material Characteristics

Much of the current research on silicon is focused on the exceedingly complex relationships among certain impurities and defects in silicon crystals, because point defects in wafer substrates exert an increasingly strong influence on final die yields. This surge of interest in crystal defect formation arises from the shrinking die and circuit-element size used in very-large-scale-integration (VLSI) technology. The complexities lie partially in the fact that impurity concentration alone does not constitute defect formation; other determining factors include thermal gradients at the time of solidification, localized melt convection, instantaneous growth rates, melt-remelt phenomena, and thermal conditions after solidification.

Current technical literature on the subject of crystal defects is plentiful, but its utilization in production practices presents many challenges. To determine accurately the influence of process changes in crystal growth upon device yields in a particular fabrication line may involve an elapsed time of three to six months, and requires detailed communication between wafer supplier and user.

Oxygen and Carbon

Control of oxygen and carbon in the growing crystal is an active area of research. Oxygen is present in CZ silicon as a result of dissolution of the silica crucible wall, and generally is higher in concentration at the top end of an ingot. Reported concentrations range from <10 to >40 ppm. Oxygen influences wafer strength, resistance to thermal warping, minority-carrier lifetime, and intrinsic impurity gettering. There is evidence that the level of oxygen content influences die yields, and that appropriate levels for maximum yield depend upon the particular device production technology.

Carbon is present in CZ silicon as a result of contributions from the original polycrystalline material and from graphite components of the hot zone. Reported concentrations range from 0.1 to 7 ppm, with levels usually rising throughout the length of the ingot. Carbon is strongly implicated in the generation of point defects; its role appears to be interrelated to the presence of oxygen.

Carbon and Oxygen Measurement/Control

Control of carbon and oxygen must be achieved primarily by the materials producer, and substantial effort is being expended in related applied research. Oxygen can be partially controlled by means of growth techniques that minimize the rate of crucible-wall dissolution and reduce the incorporation of dissolved oxygen into the growing crystal. Control of carbon is related to gas flow patterns, hot zone materials, furnace leak integrity, and starting material.

The primary technique for measuring oxygen and carbon concentrations employs a transmission/absorption-type infrared spectrophotometer. Lengthy and careful sample processing is necessary to obtain usable results, although the recent introduction of Fourier transform infrared equipment facilitates rapid measurement of oxygen and carbon on polished slices.

A subjective indication of the influence of carbon and oxygen on CZ material is provided by a variety of "swirl" tests. This technique employs high-temperature oxidation of a polished sample, followed by decoration etching and visual examination. Swirl is evidenced as a cloudy appearance, often having a swirled form, that is caused by the high-density formation of microscopic etch pits at sites where concentrated microdefects occur. Because

swirl has been correlated to areas of high device-yield loss, CZ growth technologies that provide low or zero swirl may improve die yields.

New Developments in Crystal Growing

The field of CZ growth is experiencing the emergence of several new technologies in such diverse areas as control of oxygen by magnetic fields, liquid-silicon recharging, high resistivity, and furnace automation. An overview of these areas will provide a general idea of work currently under way.

Magnetic CZ

In ordinary CZ growth, thermal gradients set up convection currents that, when combined with crucible and crystal rotation, give rise to a general flow pattern such as that in Fig. 2. This convection-induced flow transports oxygen through the melt into the liquid-crystal interface via dissolution of the quartz crucible.

The convection currents also give rise to regularly occurring temperature transients at the melt-crystal interface, creating what is termed the melt-remelt phenomenon. This repetition of incremental solidification and melting is implicated in the development of microdefects.

Standard CZ pullers afford limited capability for oxygen control through such processes as changing rotation and pull speeds and hot-zone geometry. Recently it has been found that oxygen content and crystal striations may be reduced significantly by applying a transverse magnetic field to the silicon melt area in order to damp the liquid convection currents.⁴ Several silicon

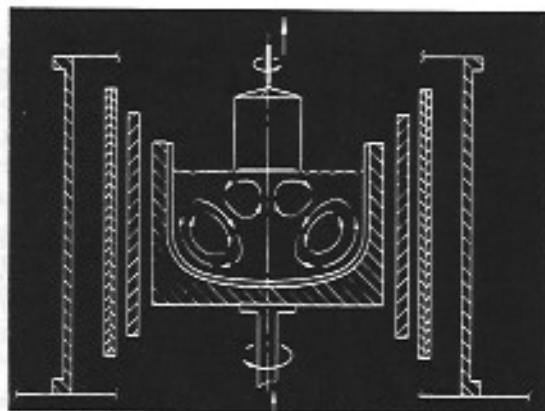


FIG. 2—Schematic representation of melt flow patterns induced by thermal convection and crucible rotation.

⁴Suzuki, T., Isawa, N., Okubo, Y., and Hashi, K. in *Semiconductor Silicon 1981*, H. R. Huff et al, Eds., Proceedings of Fourth International Symposium on Silicon Materials Science and Technology. The Electrochemical Society, Pennington, N. J., 1981.

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