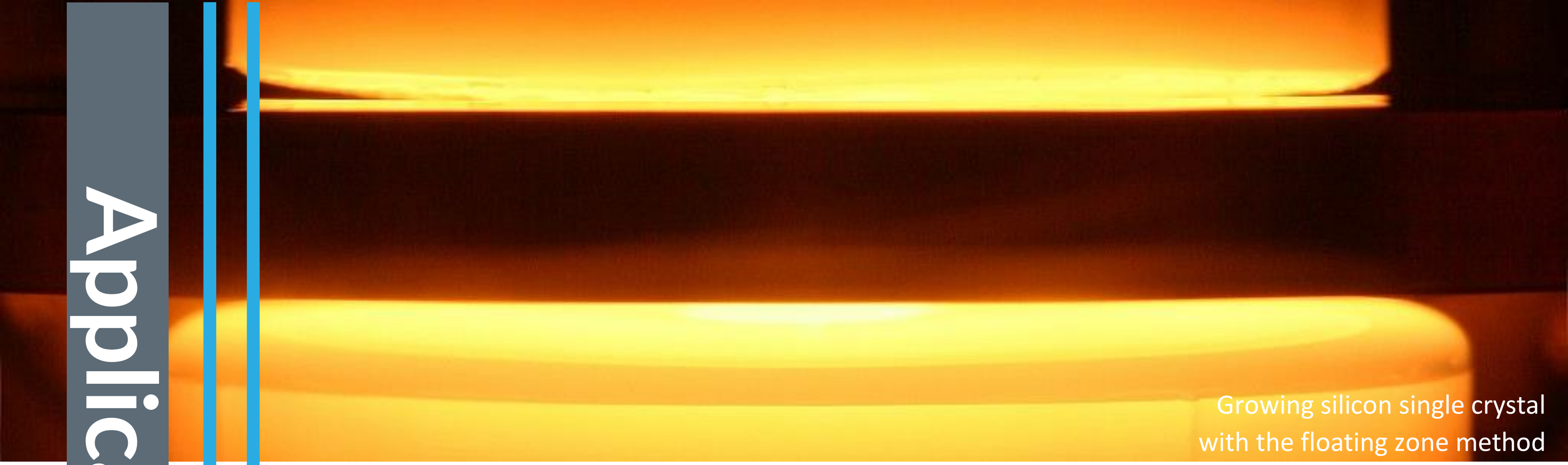
Application Note



Growing silicon single crystal with the floating zone method

Single Crystal Growth with CO_2 Laser-Heated Floating Zone

In the past driven by the semiconductor and telecommunications industries, it is today's advances in computational materials science that require flexible manufacturing technologies for the synthesis of tomorrow's crystalline materials such as ceramics, piezoelectrics, superconductors etc.

Background: As often in history, important scientific discoveries are made by accident. It is said that in 1916 the Polish chemist Jan Czochralski was investigating the crystallization of metals as he accidentally dipped his pen into molten tin instead of his inkwell and pulled out a thin tin filament.[1] Later examination of the filament revealed that it was a single crystal. Decades later, in 1948, the American scientists Gordon Teal and John Little of Bell Labs used Czochralski's method to grow extremely pure germanium and silicon single crystals, paving the way for modern semiconductor wafer production. As of today, over 90% of all semiconductor-based electronic devices are made from materials synthesized by Czochralski's method.[2]

However, only a few years later, in 1955, Henry Theuerer developed the first floating zone technique. A vertically arranged germanium rod was pulled through a localized melt zone created by an RF heating coil. The absence of a crucible allowed the growth higher purity crystals, but of limited diameter (< 200 mm). By the 1970s, the telecommunications industry was increasingly interested in growing single crystal oxides of small-diameter and high-purity due to their advantageous material properties for fiber optical applications.[3] The floating zone technique was the preferred choice but could not be applied due to inefficient RF heating of the oxides. A solution to this problem was presented in 1972 by John Haggerty, who used CO_2 laser radiation to create the floating zone in a fiber drawing process. Further improvements by Martin Fejer and Robert Feigelson in 1980 helped to establish the so-called Laser-Heated Pedestal Growth (LHPG), as shown in Figure 1.[4]

In recent years, advances in computational materials science have led to high demand for methods to exploit synthesis of new materials. The 2019 Materials by Design Roadmap reports that for the first time, the number of theoretically predicted materials exceeds the number of experimentally known entries in crystallographic databases. Thus, a shared interest by many scientists includes flexible manufacturing methods to test these newly predicted materials.[5]

Application: LHPG is such a method used in industry and material research, notably for high melting point materials, because of its flexible and cost-effective fabrication of single crystal fibers. LHPG uses focused CO_2 laser radiation to create a floating melt zone. For this purpose, the laser beam is guided into a closed chamber where it hits a reflaxicon which converts the laser beam to a hollow cylinder shape. The

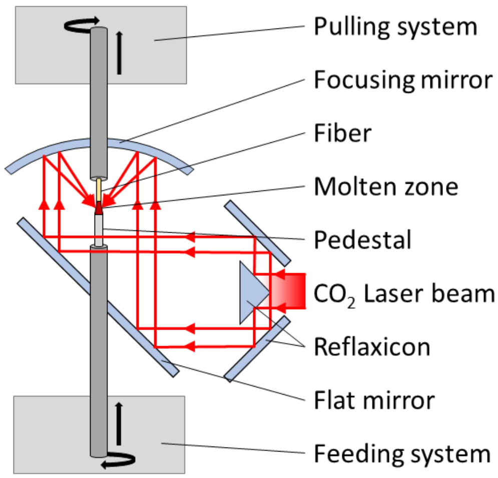


Figure 1: Drawing of the CO_2 LHPG technique.

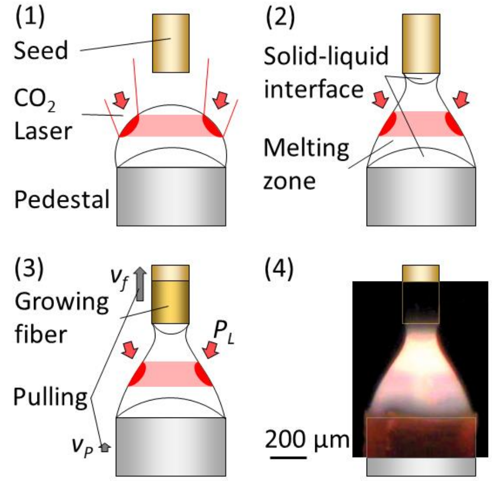


Figure 2: Drawing of the floating zone formation.

beam is then guided to a parabolic mirror which focuses the radiation over the pedestal source.[4]

The fiber drawing takes place in three steps, as shown in Figure 2. First, the focused laser radiation creates a small melt zone on top of the pedestal. Second, a seed crystal is introduced into the melt zone, creating solid-liquid interfaces at the pedestal and seed. And third, the melt zone and solidification at the seed are fed by the continuous pulling of the fiber. Under these controlled conditions, it is energetically favorable to maintain the seed's microstructure during solidification, which enables the continuous growth of the single crystal. Stable thermodynamic conditions, and thus a stable CO_2 laser source, are essential for success. In practice, however, small thermal fluctuations lead to a variation of the fiber diameter. This can be overcome by using a stabilized laser source, such as Access Laser's AL50ST, and by monitoring the melt zone (Figure 2.4). Precise control of the pulling rate v_f, v_P, and the laser power P_L can minimize the diameter variations to less than 1%.[4] The laser power required to keep the melt zone stable varies from 10 to 200 W depending on the material, diameter, pulling rate etc. Single crystal fibers with diameters from 25 µm to 1 mm have been realized.

Bottom line: Using CO_2 laser radiation as a heating source has two major advantages over other methods. First, the wavelength of 10.6 µm is well absorbed by many relevant oxides. And second, laser radiation can be easily focused, resulting in high local temperatures and gradients. The high temperature gradients enable 60 times faster growth — mm/min instead of mm/h — and the processing of materials with very high melting points. This includes materials of great interest to material science and industry such as silica, sapphire, YAG, superconductors, functional ceramics, ferro-, opto-, and piezoelectric materials, and eutectic compounds.[4] The great flexibility in producing single crystal fibers of high melting point materials at low cost makes LHPG a powerful tool.

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