3.2.5. Single Crystal Casting and Rapid Solidification

♦ Single Crystal casting

The advantage of <u>single crystal</u> growing techniques is well illustrated by describing the developments in casting of <u>gas turbine</u> blades, which are generally made of nickel-base superalloys. The procedures involved can also be used for other alloys and components.

Conventional casting of turbine blades involves investment casting using a ceramic mould. Liquid is poured into the mould and begins to solidify at the ceramic walls. The grain structure developed is polycrystalline, and the presence of grain boundaries makes this structure susceptible to creep and cracking along those boundaries under centrifugal forces at elevated temperatures.

In the **directional solidification** process, the ceramic mould is preheated by radiant heating. A water-cooled chill plate supports the mould. After the liquid is poured into the mould, crystals begin to grow at the chill-plate surface and upwards. The blade is thus directionally solidified, with longitudinal but no transverse grain boundaries. Consequently, the blade is stronger in the direction of the centrifugal forces developed in the gas turbine Fig 3.14.

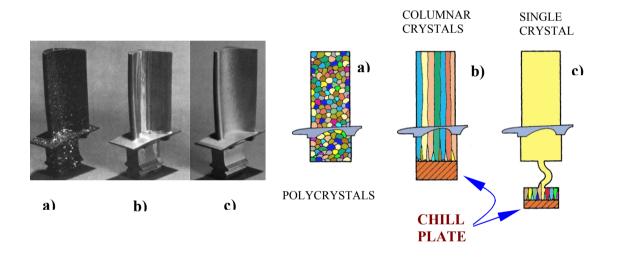


Fig 3.14: Three methods of casting turbine blades: (a) conventional casting with ceramic mould; (b) directional solidification; and (c) single-crystal blade

Single-crystal blades: The mould has a constriction in the shape of a corkscrew, the cross-section of which allows only one crystal through. A single crystal grows upward through the constriction and begins to grow in the mould. Strict control of the rate of movement is necessary. The solidified mass in the mould is a single-crystal blade. Although more expensive than other blades, the lack of grain boundaries makes these blades resistant to creep and thermal shock. Thus they have a longer and more reliable service life Fig 3.14 c).

With the advent of the semiconductor industry, **Single-crystal growing** has become a major activity in the manufacture of microelectronic devices. There are several methods of <u>crystal</u> growing that are used world-wide, e.g:

- a) <u>Seed crystal growing</u> (Crystal-pulling, <u>Czochralski process</u>)
- b) Floating-zone method
- c) <u>Hydrothermal synthesis</u> (also termed: Hydrothermal growth/method).

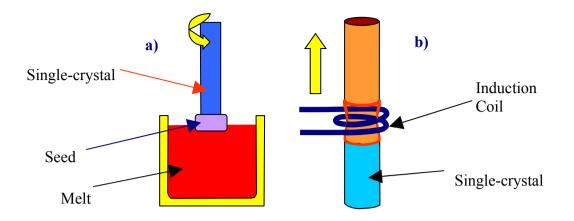


Fig 3.15: a) Crystal pulling and b) Floating-zone method

In the <u>crystal pulling</u> method, (Czochralski Crystal Growth Process shown in Fig 3.15 and 3.16) a seed crystal is dipped into the liquid and then pulled out slowly, at a rate of about $10 - 20 \mu m/s$, while being rotated at about 1 rev/s. The liquid material begins to solidify on the seed, and the crystal structure of the seed is continued throughout. *Dopants* (alloying elements) may be added to the liquid, to impart special electrical properties. This process grows single <u>crystals</u> of <u>silicon</u>, <u>germanium</u>, and various other chemical elements. Single-crystal ingots typically 50-150 mm in diameter and over 1 m in length have been produced by this technique.

A semi-metal, such as silicon is melted in a crucible in an <u>inert gas</u>. A seed crystal of the desired orientation is lowered into the liquid and the temperature is reduced slightly until the liquid begins to crystallize onto the seed. The seed crystal is then drawn up slowly, until a long, thin <u>crystal</u> (the "neck") has formed on the end.

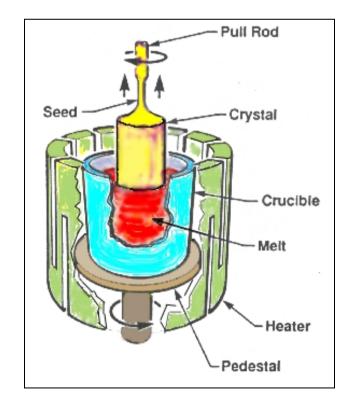


Fig 3.16: Single Crystal Growth Apparatus

Once the neck has been grown at a sufficient rate and diameter to ensure that it is free of dislocations, the temperature is reduced further and the growth rate is reduced to allow the crystal to become larger in diameter. As the diameter of the crystal approaches the desired dimension, the growth rate is again increased.

The crystal diameter is controlled during body growth by varying the lift rate of the seed and the temperature of the melt. During this period the crucible is also raised at the rate required to maintain the position of the liquid surface in relation to the heated area of the chamber. Both the seed and crucible are rotated in opposite directions and at various rates to control the properties of the crystal. The growth rate is dependent on crystal type, diameter, hot zone configuration and various other factors. For silicon, typical body diameters now range from 100 to 300 mm and typical growth rates vary from 40 to 85 mm/hr.

The single-crystal growing *floating-zone method* (Fig 3.15 b) is also widely used in the manufacture of micro-electronic devices. Starting with a rod of polycrystalline silicon resting on a single crystal, an induction coil heats these two pieces while moving slowly upward. The single crystal grows upward while maintaining its orientation. Thin <u>wafers</u> are then cut from the rod, cleaned, and polished.

<u>Hydrothermal synthesis</u> includes the various techniques of crystallizing substances from hightemperature aqueous solutions at high vapour pressures (the term "hydrothermal" originated from geochemical and mineralogical studies of hydrothermal phase equilibria).

This method of synthesis of single crystals depends on the solubility of minerals in hot water under high pressure. The crystal growth is performed in autoclaves (steel pressure vessels) in which a nutrient is supplied along with water. A gradient of temperature is maintained at the opposite ends of the growth chamber so that the hotter end dissolves the nutrient and the cooler end causes seeds to take additional growth.

Possible advantages of the <u>hydrothermal method</u> over other types of crystal growth include the ability to create crystalline phases which are not stable at the melting point. Also, materials which have a high vapour pressure near their melting points can also be grown by this method. The method is also particularly suitable for the growth of large good-quality crystals while maintaining good control over their composition. Disadvantages include the need for expensive autoclaves, good quality seeds of a fair size and the impossibility of observing the crystal as it grows.

Single crystal products include, filters, windows and sensors (e.g. for security systems and satellite equipment), antennas, laser components, scanners for brain- and heart-examination, telecommunication filters, resonators and oscillators, optical components etc. The range of industrially produced crystals includes Oxides, Tungstates, Titanates, Gallate, Aluminates, Molybdate, Zirconate, Metals and Salts.

Silicon single crystal wafers are manufactured in diameters from 25 mm - 300 mm. and used in microelectronics, solar cells and optics. GaAs single crystal wafers are made in diameters from 50 mm - 150 mm and used in microelectronics and solar cells.



♦ Rapid solidification

Rapid solidification involves cooling of a fluid at rates as high as 10^6 Kelvin/s, whereby the molten material does not have sufficient time to <u>crystallize</u> along the "long" distances. During rapid solidification <u>crystals</u> can grow at rates of up to 250 m/s however this fast growth in all directions reaches the level of the nucleation rates. Therefore the liquid solidifies into a relatively homogeneous microstructure without developing the crystalline grains. These alloys are called <u>amorphous alloys</u> or <u>metallic glasses</u> because they do not have a long-range crystalline structure. They typically contain Fe, Ni, and Cr, which are alloyed with C, P, B, Al, and Si. Among other effects, <u>rapid solidification</u> results in a significant extension of solid solubility, grain refinement, and reduced micro-segregation. Amorphous alloys exhibit excellent corrosion resistance, good ductility, and high strength. Furthermore, they exhibit very little loss from magnetic hysteresis, high resistance to eddy currents, and high permeability. The latter properties are utilized in making <u>magnetic</u> steel cores for <u>transformers</u>, generators, motors, lamp ballasts, magnetic amplifiers, and linear accelerators, with greatly improved efficiency. Another major application is superalloys of rapidly solidified powders consolidated into near-net shapes for use in aerospace engines. These alloys are produced in the form of fibre, wire, ribbon, strip, and powder.

The roll casting technique has emerged as one of the most popular rapid solidification processes for producing advanced material. Either a single or a pair of chilled rotating rolls rapidly solidifies the molten material. The single roll process is known as <u>melt spinning</u> while the process involving two rolls is termed twin-roll or strip casting. Although the two processes have physical similarity, melt spinning is most widely used in laboratories for fundamental research while twin-roll casting is primarily employed in industry.

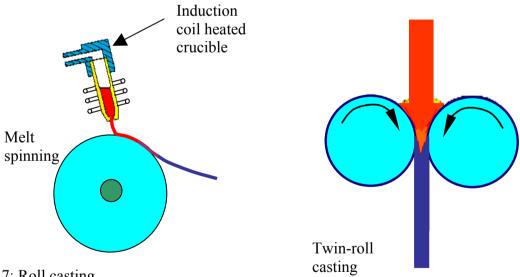


Fig 3.17: Roll casting

In melt spinning, the alloy is melted by induction in a ceramic crucible and propelled under high gas pressure at very high speed against a rotating copper disk (chill block). Consequently, the melt chills rapidly (splat cooling) and forms an amorphous solid.

A further method of rapid solidification is <u>Twin Belt Caster</u> Technique. Typical aspects of this production process include melt feeding, belt stabilization and control, heat transfer control, mould tapering, etc. Products include sheets, strips, tubes etc.

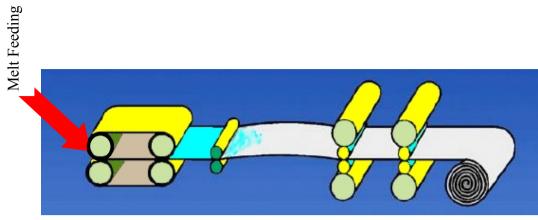
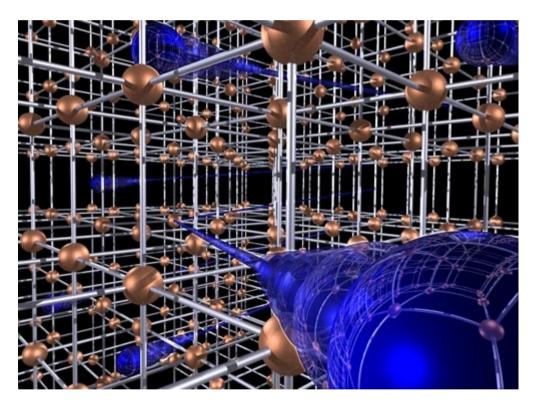


Fig 3.18: Twin Belt Casting

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Neutrons fly through a crystal lattice.

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