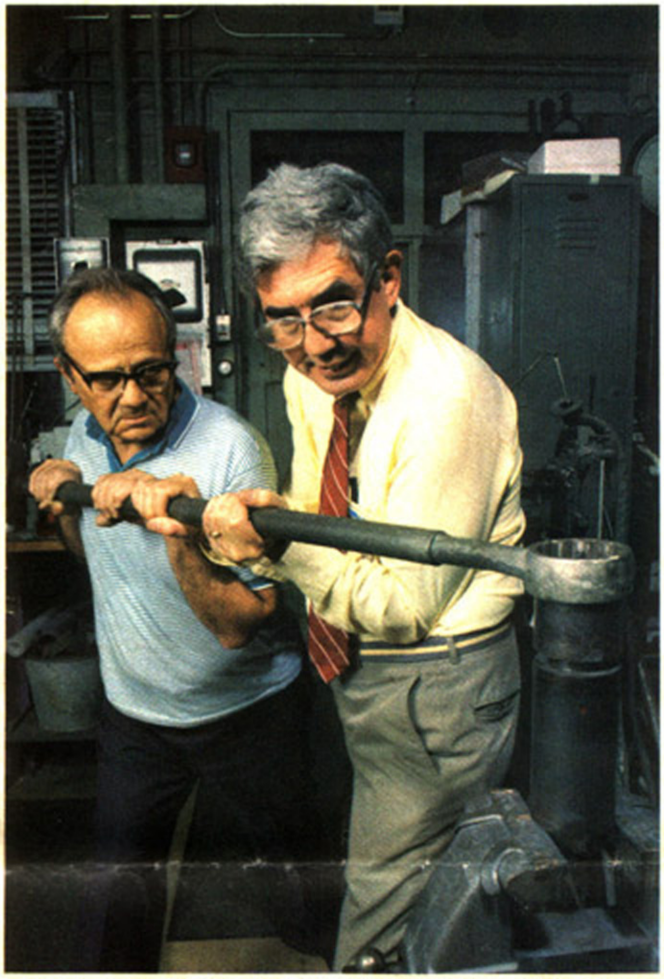

Anthony J. Caporaso (left) and Robert A. Laudise use a large wrench to open autoclave at AT&T Bell Labs

Robert A. Laudise removes furnace from a small hydrothermal autoclave prior to quenching the autoclave

Institute of Crystallography of the Soviet Academy of Sciences. Studies at Bell Labs show that to produce DLF quartz, DLF seeds must be used and special care taken to avoid inclusions of solid particles, which often are corrosion products formed by reaction with the steel walls of the autoclave. Such inclusions lead to strain and often dislocations when they are covered over by the growing crystal. Present research focuses on further perfecting crystal purity and physical properties, which affect the performance of advanced piezoelectric devices.

Aluminum phosphate

Because it has a larger piezoelectric coupling constant than quartz, single crystal aluminum phosphate ($AlPO_4$) is of interest as a material for electronic devices, especially surface acoustic wave devices. This has led to worldwide research on growing aluminum phosphate crystals in the past few years.

Aluminum orthophosphate (berlinite) is isostructural and isoelectronic with quartz. In it, one group III Al and one group V P substitute for two group IV Si atoms. The alpha form is stable below about 584 °C. This suggests that hydrothermal growth techniques are appropriate for growing $AlPO_4$ crystals.

Most of the methods now used are based on the work of Joseph Stanley at the U.S. Army Electronic Technology & Devices Laboratory at Fort Monmouth, N.J., who discovered that $AlPO_4$ has appreciable solubility in phosphoric acid but that the temperature coefficient of solubility is usually negative. He and his colleagues generally grew crystals by slowly heating saturated solutions. Subsequent work by William R. McBride at the Naval Weapons Center, China Lake, Calif., has extended Stanley's techniques, providing larger crystals for device evaluation. Studies by Ernest Kolb and me at Bell Labs have been aimed at perfecting a continuous high-growth-rate process.

Crystals of $AlPO_4$ are difficult to grow largely because of its negative temperature coefficient at temperatures below 300 °C, where its solubility is large enough to permit growth. Thus, in the usual hydrothermal temperature gradient, in which the bottom of the autoclave is hotter than the top and convection is effective, $AlPO_4$ would be expected to nucleate in the bottom region. Consequently, the seed is placed in the hotter lower region and the nutrient in the cooler top region. This approach has significant advantages. When the bottom of the autoclave is warmer, induced convection provides sufficient mixing so that growth rates are almost never limited by transport except for diffusion near the growing crystal interfaces. This seed-at-the-bottom geometry requires that the particles of nutrient be large enough to remain in a relatively open mesh basket in the upper region of the autoclave.

Bruce H. T. Chai of Allied-Signal Research Laboratory in Morristown, N.J., has prepared $AlPO_4$ crystals using large (60 mesh) particles of nutrient in kilogram quantities. He typically heats and reacts aluminum hydroxide with excess phosphoric acid in an autoclave filled so that the pressure inside it is on the vapor pressure curve, cycling the temperature daily between