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Abstract

Single crystal fibers of four refractory oxide materials (Al_2O_3 , $\text{Cr}:\text{Al}_2\text{O}_3$, $\text{Nd}:\text{YAG}$ and LiNbO_3) have been grown by a miniature pedestal growth technique. The growth apparatus employs novel electronic control, mechanical and optical systems enabling growth of high optical quality fibers. All four materials exhibit similar growth characteristics and yield fibers of comparable quality. Measured optical waveguide losses at 632.8 nm for a 5 cm long 170 μm diameter $\text{Cr}:\text{Al}_2\text{O}_3$ fiber were 0.04 dB/cm.

Introduction

The ability to fabricate optical waveguides in materials other than glass offers new and interesting device opportunities. In particular, waveguides formed from single crystal fibers offer the potential to make devices that utilize the unique optical and nonlinear optical properties of single crystals in a guided wave structure.

In this paper we report the growth of single crystal fibers using a laser assisted miniature pedestal growth technique.^{1,2,3} To implement the growth of small diameter, oriented, single crystal fibers, we have designed and constructed a fiber growth apparatus that uses a waveguide CO_2 laser source and a unique symmetrical optical focusing system.^{4,5}

The advantages of single crystal fibers are best illustrated by considering potential applications. The applications, in turn, generate a set of criteria that the single crystal fibers must meet if they are to be useful in devices. Following the discussion of applications and fiber parameters, we describe the growth apparatus and recent growth results. We then review optical measurements of single crystal fibers and summarize progress toward single crystal fiber devices.

Applications and properties of single crystal fibers

The growth of single crystal fibers is motivated by their application to linear and non-linear optical devices that are not possible in glass fibers. For convenience, we classify devices as passive, active and nonlinear. Table I lists examples of possible single crystal fiber applications within these classes and illustrates each application area by some representative materials. In addition to device applications, the rapid growth of single crystal fibers makes them useful for material surveys.

Table I. List of 4 different classes of applications and some specific devices in each class. Some representative crystalline materials for each application are given in the right hand column.

Passive Devices	
Lightguide	Al_2O_3
Thermometer	
Polarizer	
Active Devices	
Laser amplifier	$\text{Nd}^{+++}:\text{YAG}$
Laser oscillator	$\text{Cr}^{+++}:\text{Al}_2\text{O}_3$
Nonlinear Devices	$\text{Ti}^{+++}:\text{Al}_2\text{O}_3$
Modulator	LiNbO_3
Mixer	
Harmonic generator	
Parametric oscillator	
Materials Surveys	
Laser host-ion combinations	$\text{Ti}:\text{Al}_2\text{O}_3$

To date we have grown single crystal fibers of Al_2O_3 , $\text{Cr}^{+++}:\text{Al}_2\text{O}_3$, $\text{Nd}^{+++}:\text{YAG}$ and LiNbO_3 , in the orientations, lengths, and diameters shown in Table II.. This list is representative and not intended to be complete, since more than thirty materials have been grown by this technique at Stanford University.⁶

The optical attenuation of a single crystal fiber is determined by a number of factors including crystal defects, impurity concentration, compositional inhomogenieties and diameter fluctuations.

Fiber diameter control is of concern since random diameter fluctuations of only 1% can lead to modal conversion and scatter losses in the range of $5\% \text{ cm}^{-1}$. However, for a typical case, diameter fluctuations of 0.1% yield calculated losses of only $0.05\% \text{ cm}^{-1}$. Periodic diameter modulation of

the fiber also leads to scatter losses. However, if properly controlled, periodic fiber diameter modulation may be useful as a Bragg mirror or filter.

Table II. Representative Single Crystal Fibers

Material	Orient- ation	Length (cm)	Diameter (μm)
Al_2O_3	<001>	20	170
		3.5	50
$\text{Al}_2\text{O}_3 + 0.05 \text{ wt } \% \text{ Cr}$	<001>	10.0	170
		3.0	95
YAG + 0.9 wt % Nd	<111>	3.5	110
LiNbO_3	<001> <100>	3.5	50
		3.0	170

The optical loss resulting from diameter variations can be reduced by using a diffused cladding if the diffusion depth is large compared to the scale length of the diameter variations. Such a cladding might be achieved in LiNbO_3 by diffusing protons into the fiber using known techniques.⁷

To date fibers grown with our apparatus have shown diameter fluctuations on the order of several percent under open loop growth conditions. We have designed and plan to implement closed loop diameter control during fiber growth

using a novel diameter measurement apparatus.⁸ Fiber diameter uniformity of 0.1% should be achieved using this system.

The device applications of single crystal fibers shown in Table I determine the fiber parameters necessary for good device performance. Important crystal fiber parameters include length, diameter, and optical loss. Table III summarizes typical single crystal parameters required for thermometry using an Al_2O_3 fiber⁹ and for second harmonic generation using a LiNbO_3 fiber.

Table III. Typical single crystal fiber parameters for device applications

Passive device	
(Thermometry using sapphire)	
length	- 20 cm
diameter	- 70 microns
diameter variations	- 1%
Nonlinear device	
(SHG using lithium niobate)	
length	- 5 cm
diameter	- 25 microns
diameter variations	- 0.1%
efficiency	- 0.1%/mW

High temperature thermometry is possible using blackbody emission guided along a sapphire fiber. Since the sapphire fiber can be optically coupled to a glass fiber only the portion of fiber exposed to high temperatures need be sapphire. We thus anticipate relatively short, e.g. 20 cm, fiber lengths to be useful. Constraints on fiber flexibility generally determine the maximum allowable fiber diameter. A 70 μm diameter sapphire fiber can be readily bent on a 1 cm radius of curvature, adequate for most applications.

This device is not particularly sensitive to optical loss or mode coupling within the fiber. The allowable diameter variations are, therefore, relatively loose, on the order of 1%.

To date high temperature sapphire thermometry has made use of short sapphire rods coated with iridium metal at the tip.⁹ Recently we have grown 170 μm diameter fibers of sapphire up to 20 cm in length for this application. We have also successfully doped the end of the sapphire fiber with metal to provide an integral, stable, blackbody source. Tests are now in progress to demonstrate the use of sapphire fibers as a flexible, high temperature, high speed thermometer.

Nonlinear devices using crystal fibers promise higher frequency conversion efficiency than conventional devices since fibers allow a tight beam confinement over a long interaction length. For example a 5 cm long 25 μm diameter LiNbO_3 fiber offers a factor of 50 efficiency improvement compared to second harmonic generation in a bulk crystal. Since this device requires low loss single mode optical propagation in the fiber, the diameter control tolerances are tight, on the order of 0.1%. Thus high conversion efficiency is possible with incident powers on the order of 100 mW, which is the power level now available in single mode diode laser sources.

Crystal growth apparatus

We have designed and built a growth apparatus to produce single crystal fibers of the quality required for device applications. The apparatus uses a laser heated miniature pedestal growth technique first applied to optical fiber growth by Burrus and Stone.² Figure 1 illustrates growth of a single crystal fiber using the laser heated miniature pedestal growth technique.

A waveguide CO_2 laser is focused onto the molten zone by a combination refraxicon/parabolic mirror optical system shown schematically in Fig. 2. A source rod is translated into the focused laser beam via a belt drive translation system. The source rod may be

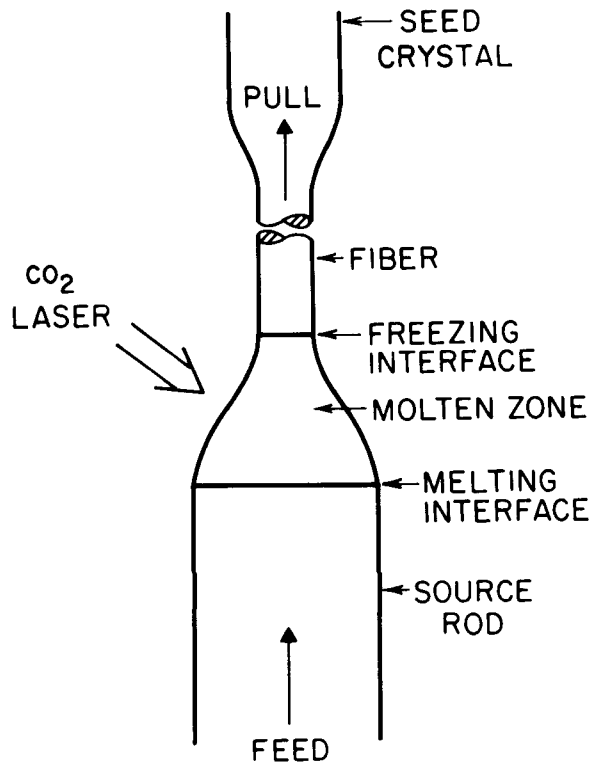


Figure 1. Schematic of miniature pedestal growth.

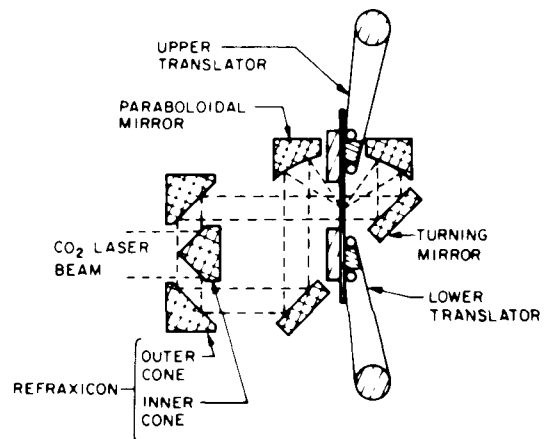


Figure 2. Schematic of focusing system and fiber translation devices.

fabricated from either single crystal, polycrystalline, or hot pressed powder material.

To initiate growth an oriented seed rod is dipped into the molten zone. The seed rod defines the crystallographic orientation of the fiber. Growth proceeds by simultaneously translating the lower and upper fiber source and seed rods. Conservation of mass determines the fiber diameter reduction as the square root of the feed rate to pull rate ratio. Diameter reductions of 3:1 are typical. Greater diameter reductions are difficult due to the onset of growth instabilities.

Miniature pedestal growth differs from the viscous drawdown of a glass fiber since, unlike glass, crystals have a definite solid/liquid phase transition. The molten zone is a true liquid being held in place by surface tension.

In order to achieve a stable fiber diameter, stable fiber growth conditions must be realized. This in turn dictates a stable mechanical apparatus, smooth feed and pull rates, stable laser power and symmetric heat input into the molten zone.

The optical system shown schematically in Fig. 2 uses copper mirrors to focus the CO₂ laser source onto the molten zone. The refraxicon, in combination with the f/2 focusing parabolic mirror, yields a symmetric focus with a 30 μm diameter. The tight focus allows the growth of small diameter fibers.

The translation system shown in Fig. 2 uses a seamless-belt drive system driven by a phaselocked dc motor. The fiber is held in a 'V-groove' etched in a silicon substrate that is oxidized to form a hard silicon dioxide surface. This drive system is in turn controlled by a digital logic system that allows control of the growth rate and diameter reduction ratio.

The present growth apparatus yields fibers with 2% diameter variations over centimeter lengths. The growth of more uniform fibers will require active control of the fiber diameter during growth. Figure 3 shows a schematic of the fiber growth apparatus including a fiber diameter measurement and control system. Since no commercial fiber diameter system met our sensitivity, speed, and working distance requirements, we have designed and built the non-contact diameter measurement system shown in Fig. 4.

The fiber diameter measurement system uses a helium-neon laser to illuminate the fiber. The beam is tightly focused using a cylindrical lens to define the measurement zone along the fiber. In the other plane the beam is scattered by the fiber and imaged onto a

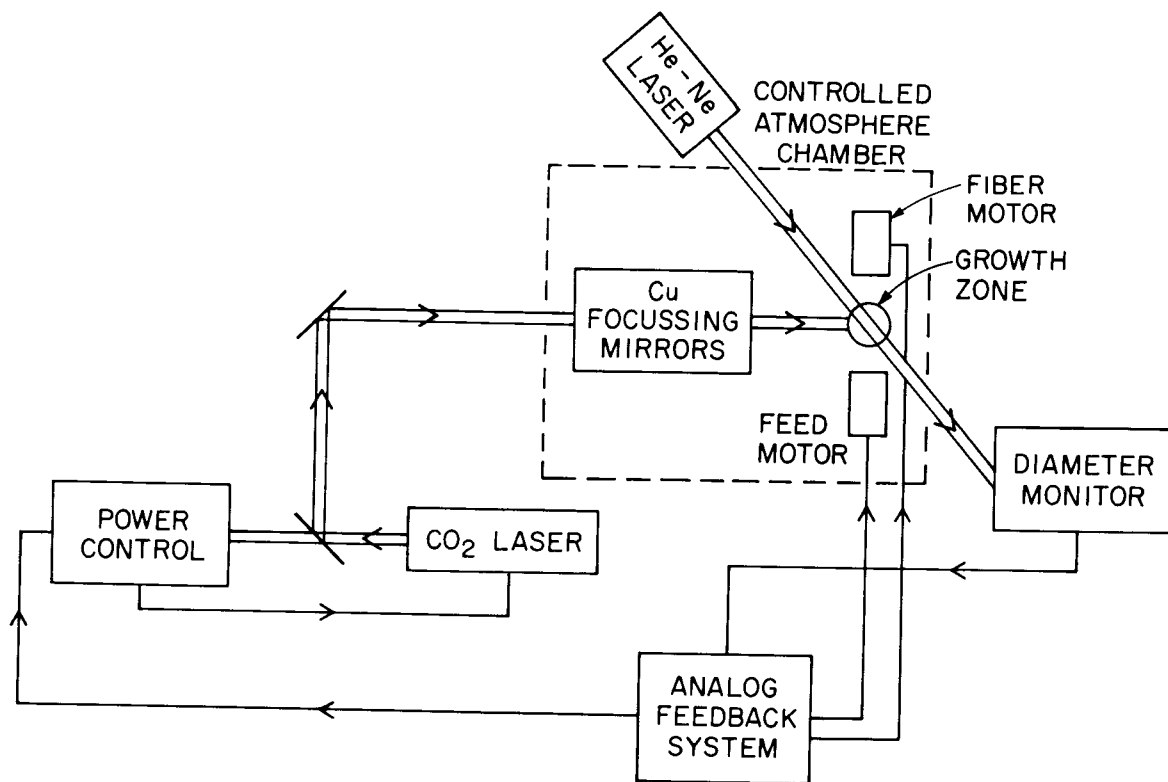


Figure 3. Schematic of fiber growth apparatus.

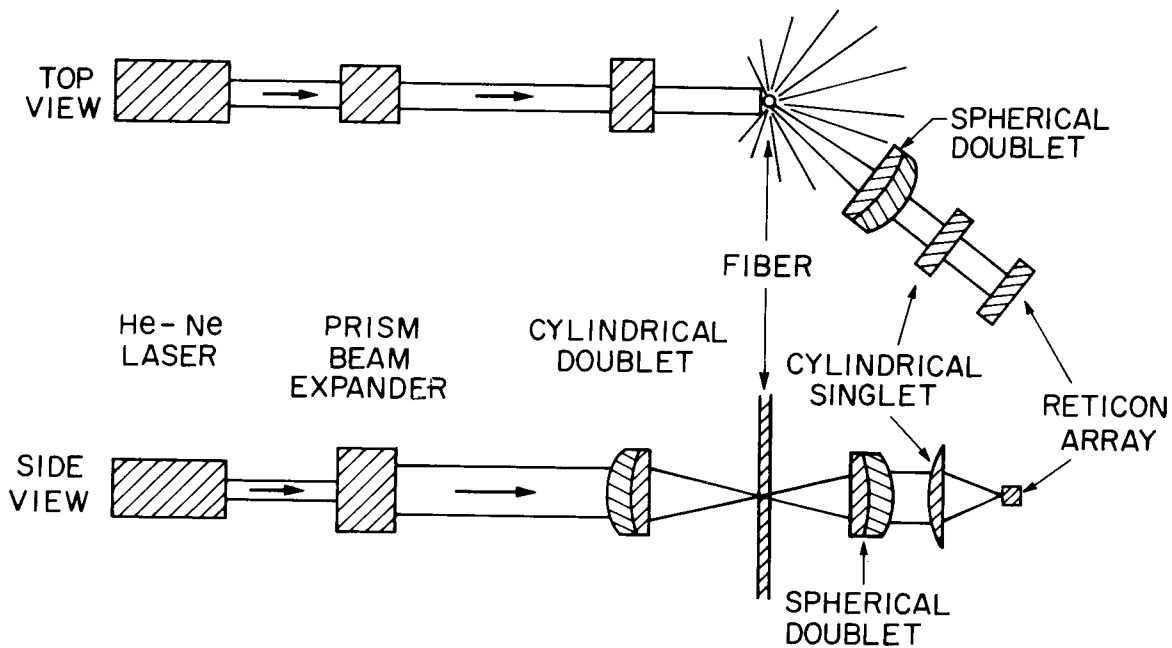


Figure 4. Schematic of diameter measurement system.

a photodiode array where interference fringes are detected.¹⁰ The interference pattern is analyzed to provide an output signal proportional to the fiber diameter. Recent measurements have shown that the diameter sensitivity is $\pm 500 \text{ \AA}$ at a measurement rate of 1 kHz.

The diameter measurement system has not yet been incorporated into the growth apparatus. Thus all fibers grown to date have been without diameter feedback control.

Fiber growth results

In the six months that the fiber growth apparatus has been operational four crystalline materials have been grown: sapphire, ruby, Nd:YAG and LiNbO_3 . Table II summarizes the length, diameter and orientation of the single crystal fibers. We initiated our growth studies by concentrating on sapphire and chromium doped sapphire or ruby because of availability and ease of growth. Experience with sapphire has enabled us to extend growth studies to Nd:YAG and to LiNbO_3 . To date we have grown both a-axis and c-axis LiNbO_3 with diameters as small as 50 \mu m and lengths to 3.5 cm.

Typical fiber growth rates range between 1 and 10 mm/min. While these growth rates are slow compared to glass fiber pulling rates, they are orders of magnitude faster than bulk crystal Czochralski growth rates. Since fiber lengths of 5 cm are adequate for many applications useful fiber lengths can be grown in approximately twenty minutes.

The growth apparatus is designed to yield fiber diameters of between 500 and 20 \mu m . However, smaller diameter fibers to 6 \mu m have been grown under special circumstances.⁶ The initial feed rods are fabricated to 500 \mu m diameter using a centerless grinder. Diameter reductions of 3 to 1 are normally used during growth. Smaller diameter fibers are grown using previously grown fibers as source rods. The CO_2 laser power required to grow Al_2O_3 and LiNbO_3 fibers from 500 \mu m diameter feed rods are 4.8 and 1.5 watts respectively. The difference is chiefly due to the different melting temperatures of the materials, 2045°C for sapphire and 1260°C for LiNbO_3 . The required laser power reduces to less than 1 W for fibers grown from 170 \mu m diameter feed rods.

Sapphire, Nd:YAG and LiNbO_3 all display similar growth characteristics. Figure 5 shows the molten zone shape for Al_2O_3 . The molten zone shape is similar for the other materials. For optimum growth stability the laser power is adjusted to yield a molten zone with a height-to-width ratio of near unity for a 3 : 1 diameter reduction ratio.

The single crystal fiber growth morphology is similar to that seen in bulk Czochralski growth. For example, $\langle 111 \rangle$ axis Nd:YAG fibers show a slightly rounded hexagonal cross-section as shown in Fig. 6. LiNbO_3 grown along the optic axis shows a characteristic 3-fold symmetry with growth ridges running parallel to the crystal length as shown in Fig.7.

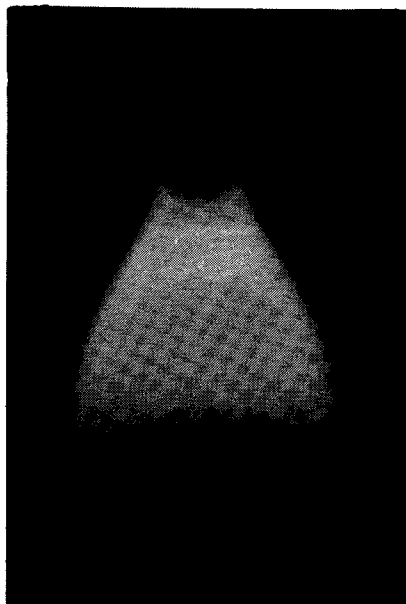


Figure 5. Photomicrograph showing growth of a 170 \mu m diameter Al_2O_3 fiber from a 500 \mu m diameter source rod. The growing fiber is invisible since its smooth sides scatter very little light.

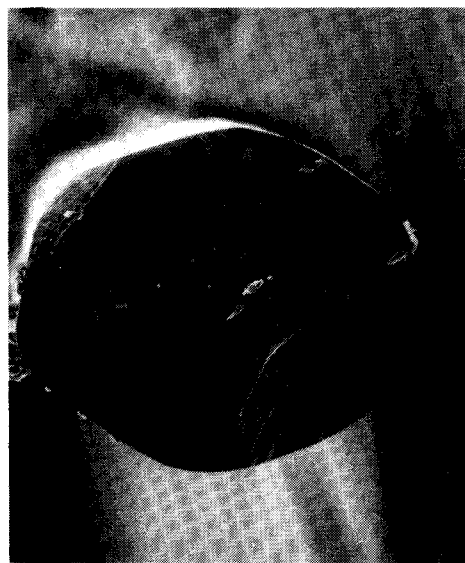


Figure 6. Scanning electron microscope photograph of cross-section of Nd:YAG fiber.

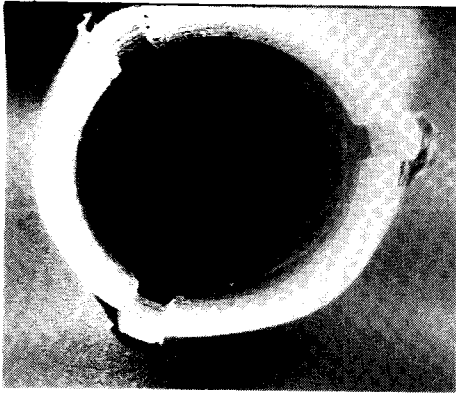


Figure 7a. Scanning electron microscope photograph of cross-section of LiNbO_3 fiber showing pronounced 3-fold growth ridges. The internal concentric ring pattern and dark central area are artifacts of the procedure used to obtain the cross-section.

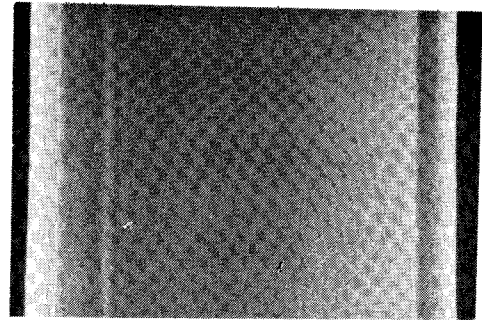


Figure 7b. Scanning electron microscope photograph of surface of a LiNbO_3 fiber. Note the sharply defined growth ridges at the right boundary and near the left edge.

Since the fiber cross-section is invariant with length, the growth morphology should not be detrimental to optical applications.

Early single crystal fibers showed fine scale diameter fluctuations along the fiber length with a scale of 1 - 10 μm .⁴ Crystals grown with the current machine are free of these irregularities as shown in Fig. 7b. At a longer length scale we have demonstrated diameter stability to 2% over fibers several centimeters in length.

Optical measurements

We recently have initiated optical property measurements on single crystal fibers. For example, we have measured an optical waveguide loss of 0.04 dB/cm at 632.8 nm in a 5 cm long 170 μm diameter ruby fiber. Comparable losses have been measured in other fibers. For these measurements the single crystal fibers were not cladded so that guiding was provided by the crystal-air dielectric interface. We anticipate lower propagation losses as we grow improved optical fibers and employ more refined cladding techniques.

Sapphire fibers as now grown are suitable for some light-guide applications such as thermometry. For example, we have demonstrated optical propagation in a sapphire fiber heated to 1500°C with no significant change in transmission at the elevated temperatures.

Summary

In summary we have designed and built a fiber growth apparatus to produce high quality single crystal fibers. The apparatus uses the miniature pedestal growth technique which permits the growth of crystal fibers in a wide range of materials. The apparatus is designed to handle fiber diameters as small as 20 microns with fiber lengths exceeding 20 cm. Diameter stability to 2% has been demonstrated over fiber lengths of several centimeters. The optical attenuation of these fibers is 0.04 dB/cm. These fibers are of sufficient quality for simple passive device applications such as high temperature thermometry.

A fiber diameter measurement system has been built which when installed in the growth apparatus should improve the diameter stability and the optical attenuation by an order of magnitude. We anticipate these fibers will be of sufficient quality for a host of active and nonlinear device applications.

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