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SIZING SINGLE CRYSTAL SAPPHIRE

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ABSTRACT

Although there are several significant advantages to using single crystal sapphire as substrate material for hybrid microcircuits, the sizing of this material can be far more troublesome than the sizing of alternate material, such as polycrystal alumina. This report describes the work done in developing a satisfactory method of sizing single crystal sapphire. It gives the results of sizing with grinding wheels, wire saws, and laser scribers. It was concluded that laser scribing is a viable method of sizing if the sapphire is grown parallel to the r plane. This takes advantage of the easy crystal cleavage planes and reduces the risk of edge chipping and nonperpendicular breaking.

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SIZING SINGLE CRYSTAL SAPPHIRE

Introduction

Substrates are necessary to provide both the mechanical support and the electrical insulation for metal films used in hybrid microcircuits. Various materials are used to fabricate these substrates, depending on the requirements for a particular application. Ideally, a substrate should provide mechanical strength; good electrical isolation; a flat, smooth surface; high heat conductivity; and good adhesion for the metallization.

In the past, Sandia Laboratories found that "as-fired" alumina met the minimum requirements for the existing programs. In addition, alumina is readily available, reasonably priced ($.10/2.54 cm x 2.54 cm x 0.064 cm and easily sized. Hybrid microcircuit trends toward increased circuit density and multilayer metallization may render the alumina inadequate as a substrate material because of surface roughness. The new high density line and space definition (0.0254 mm lines and 0.0245 mm spaces) requires the best surface finish available. Any imperfection in surface conditions causes photolithography and subsequent deposition problems.

Sapphire approaches all the mechanical, chemical and electrical characteristics needed by Sandia Laboratories in anticipated high circuit density applications; in addition, its surface can be polished to a very fine finish (.0001 μin/in) more readily than polycrystaline alumina can be polished.

Material Comparison

There is no one substrate material that has the best of all the desired characteristics. The weight placed on each property depends on the program in which the substrate will be used. A list[1] of the ideal substrate properties is shown in Table 1. In addition, characteristics such as repeatability, low parasitic capacitance, and transparency for back side inspection are often considerations.

TABLE 1

Desired Properties

ATOMICALLY SMOOTH SURFACE
PERFECT FLATNESS
NO POROSITY
MECHANICAL STRENGTH
THERMAL COEFFICIENT OF EXPANSION EQUAL TO THAT OF DEPOSITED FILM
HIGH THERMAL CONDUCTIVITY
RESISTANCE TO THERMAL SHOCK
THERMAL STABILITY
CHEMICAL STABILITY
HIGH ELECTRICAL RESISTANCE
LOW COST

With the possible forthcoming requirement of 0.0254 mm conductor lines and 0.0254 mm spaces, it became apparent that a substrate with exceptional surface smoothness would be needed. Commercial two-sided optically smooth sapphire substrates were procured and an extensive evaluation[2] was performed to determine feasibility of using this type substrate in hybrid applications. As seen in Table II[3], the properties of sapphire compare favorably with other standard substrate material.

TABLE II

Properties of Substrate Materials

Property	Fine Grained Alumina	Coarser Grained Alumina	Glazed Alumina	Glass (7059)	Polished Alumina	Polished Sapphire	Units
Surface Smoothness	3-4	8-10	1-2	1	1	1	μ inch CLA
Flatness (Camber)	2	4	4	4	.5	.5	MIL/inch
Flexural Strength	90	70	60	20	60	90	x 10^3 psi
Thermal Conductivity	.38	.38	~.1	.0125	.38	.41	watts/cm°C
Thermal Expansion	6	6	6.4	4.5	6	6.6* 5.0	ppm/°C
Softening Point	1500	1600	900	872	1600	1800	°C
Resistivity (300°C)	$>10^{10}$	$>10^{10}$	$>10^{9}$	10^{12}	$>10^{10}$	$>10^{10}$	ohm-cm
Dielectric Constant	9.8	10	8.9	5.8	9.8	11.5** 9.4	at 1MHz
Loss Tangent	<.0001	<.0001	.0005	.0011	<.0002	<.0001	at 1MHz
Diamond Scribability	✓	NO	NO	✓	NO	NO	
Laser Scribability	✓	✓	✓	✓	✓	✓	

*6.6 // C-Axis
5.0 ⊥ C-Axis

**11.5 // to C-Axis
9.4 ⊥ to C-Axis

Material Used

The sapphire used in the initial evaluation had the material properties shown in Table III.[4] The single crystal material was grown perpendicular to the c face (0001 plane). Figure 1 is a drawing of a typical Al_2O_3 rhombohedral crystal showing the c face. The substrates were 25.4 mm x 38.1 mm x 0.381 mm. The surface was defined as scratch-free on both sides and had a smoothness of approximately .0254 μm center line average. The substrates were transparent to allow optical inspection of the deposited metallization from the back side. A visual inspection of the edges showed that they had been lapped or ground as a last sizing step.

TABLE III

Sapphire Characteristics

PHYSICAL FORM	Rhombohedral single crystal
DENSITY	3.97 grams per cubic centimeter: 0.143 pounds per cubic inch
HARDNESS	2000 kilograms per square millimeter-- 9 MOHS hardness scale; quartz is 7, diamond is 10
TENSILE STRENGTH	Minimum of 350,000 pounds per square inch for 0.010 diameter filament at room temperature
COMPRESSIVE STRENGTH	450,000 psi at 25°C
COEFFICIENT OF FRICTION (against steel)	0.15
SPECIFIC HEAT	0.18 calories per gram at 20°C
MELTING POINT	2053°C. 3727°F
MODULUS OF ELASTICITY	67.5×10^6 pounds per square inch
THERMAL CONDUCTIVITY	0.1 Calorie/centimeter-second-°C at 25°C 24 BTU/hour-foot-°F
THERMAL EXPANSION	9.3×10^{-6}/°C parallel to C-axis 8.8×10^{-6}/°C parallel to A-axis Average values from room temperature to 1100°C
EMITTANCE (λ=2.6-3.7 microns)	Less than 0.02 measured (at 880°C)
RESISTIVITY	1×10^{16} OHM-centimeter at room temperature 1×10^{11} OHM-centimeter at 500°C
DIELECTRIC CONSTANT	11.0 parallel to C-axis 9.3 parallel to A-axis At 25°C between 10^3 Hz and 10^{10} Hz
DIELECTRIC STRENGTH	480,000 volts/cm at 60 Hz
DIELECTRIC LOSS TANGENT	0.00003 C-plane, 10^{10} Hz at 25°C 0.000086 A-plane, 10^{10} Hz at 25°C
EXPOSURE TO GAMMA RADIATION	No visible color after exposure to 10^3 Rads/hr for 60 minutes at -195°C. No change in transmission above 2.5 microns after exposure to 10×10^6 Rads
EXPOSURE TO PROTRON RADIATION	No change in transmission below 3000A after exposure to 10^{12} protrons/cm^2 total dose
OPTICAL TRANSMISSION	Up to 98.5% total transmission as measured by the integrating sphere method
INDEX OF REFRACTION	(Sodium D line) birefringent; No=1.768; Ne=1.760

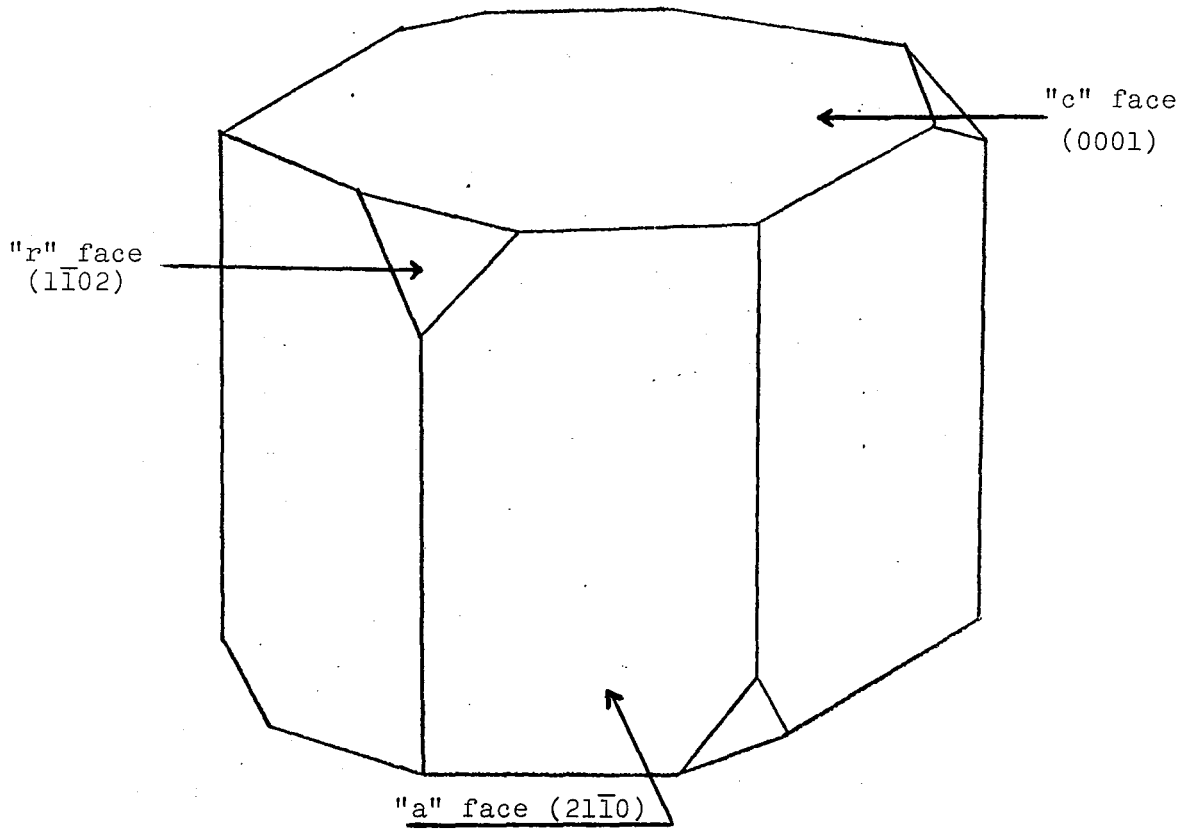


Figure 1. Corundum: Al_2O_3 Rhombohedral sym.
Cleavage (easy) parallel to "r" face.

Methods of Sizing

Because of encouraging results in the metallization evaluation of sapphire,[2] sapphire was chosen as the substrate material used in the hybrids of the Secure PAL program. Up to that time, the only significant disadvantage known about sapphire was its high cost, $40.00/50.8 mm x 50.8 mm x .381 mm. After a multilayer metallization pattern was deposited (Figure 2) on the single crystal sapphire, the next step was to separate the individual patterns by sizing. The standard method at Sandia Laboratories for sizing polycrystaline alumina substrates is to scribe using the neodymium YAG laser (Figure 3), and to hand break along the scribe lines.[5] When this was attempted with sapphire, it appeared that the optically finished sapphire seemed to be nearly transparent to the laser beam, and an adequate scribe could not be accomplished.

The next attempt at sizing the sapphire was to use a diamond blade on a circular power saw. The cut was first attempted on a single substrate. The cut was ragged and pulled out chips approximately .229 mm into the substrate (Figure 4). Next, a substrate stacking approach was used. A sapphire substrate was stacked between two glass substrates; the sandwich was held together using glycol, and sawed. The saw edges were much cleaner, but there were still areas that took as much as .153 mm chips out of the surface. In the particular application that the first sapphire substrates were to be used, the maximum substrate width was 4.6 mm. The active pattern on the substrate was 4.2 mm wide. With .19 mm on either side of the substrate from the edge to the beginning of the metallized pattern, there would undoubtedly be a percentage of substrates lost because of these chips and also because of microcracks that develop at stress points in the chipped areas.

Another method used to size the sapphire was a 0.2 mm diameter beryllium copper wire saw. The wire was 30.5 m long, and the cutting was actually accomplished by an abrasive slurry of 240 grit silicon carbide

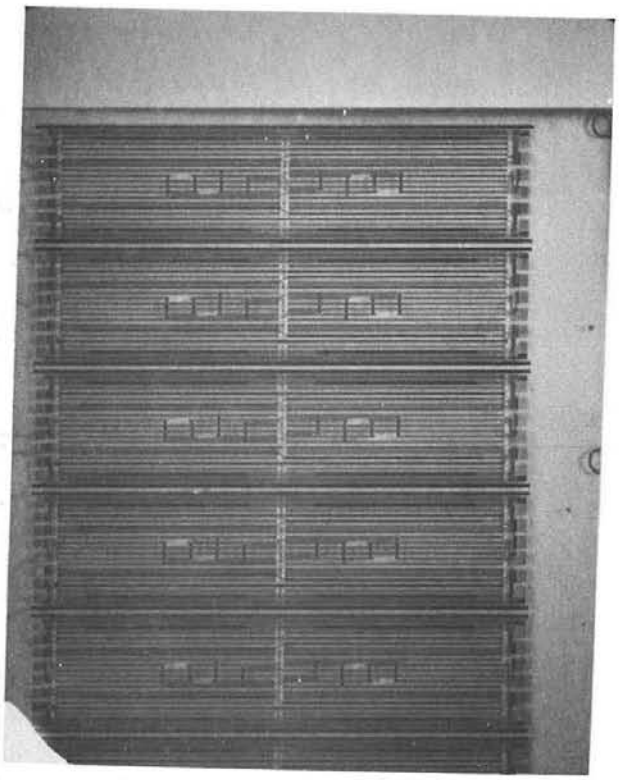


Figure 2. Gang Multiple Layer Metallization Pattern

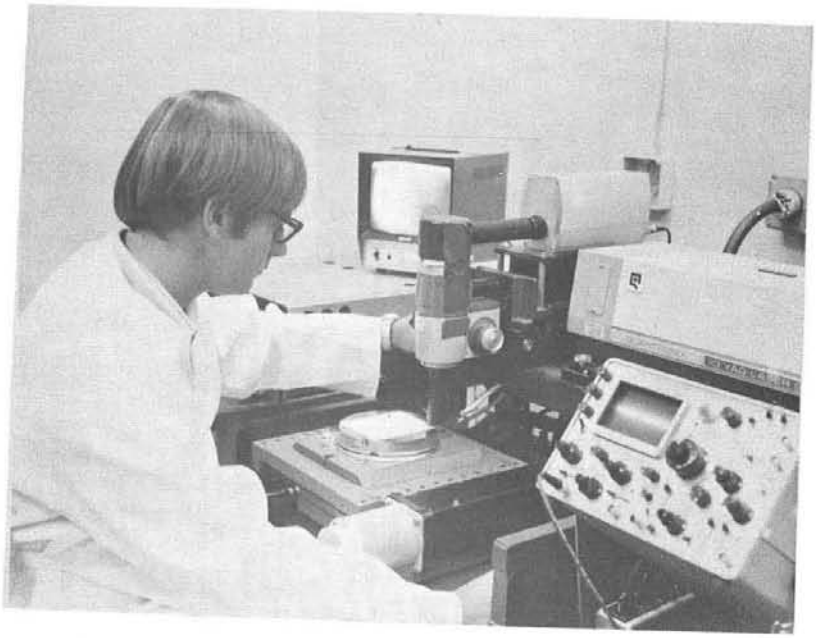
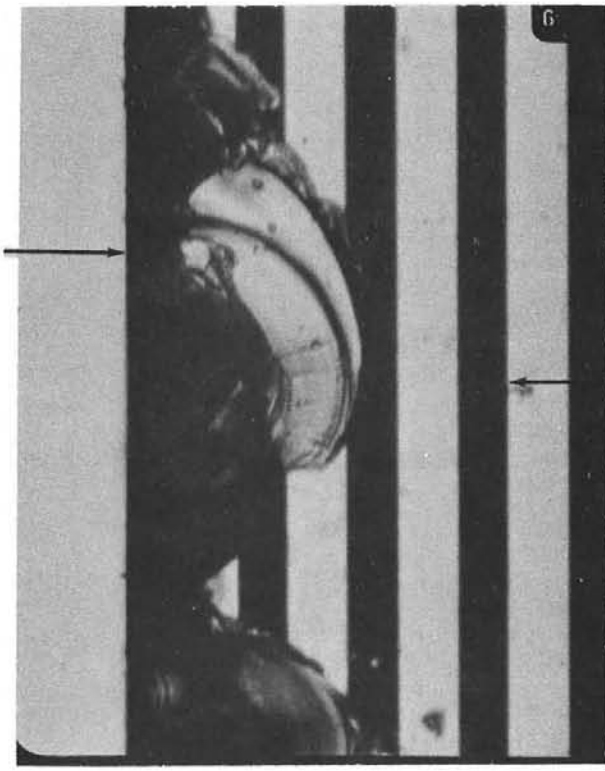


Figure 3. Laser Scribing Operation



100 X Sapphire Saw Edge

Figure 4. Large Chips Caused by Saw

in #10 vehicle. The resulting cut was relatively clean with no evidence of chipping. However, each cut took five hours and therefore was not practical for production consideration.

Ed Machin[6] suggested that the original YAG laser could be used if enough care was taken in focusing the beam and by slowing down the speed of the beam across the substrate. His first attempts did scribe the sapphire, and the breaking followed the scribe lines. However, when the profile of the sized edge was examined under 80X magnification, Figure 5, it could be seen that two of the scribed edges located opposite each other appeared to be broken along approximately a 30° direction (rather than 90°) to the metallized surface. The other two edges broke at 90°. The 30° angle caused a problem by increasing the overall width of the substrate by .30 mm (2 x cotan 30° with a .35 mm thick substrate, with the scribe depth approximately half the substrate thickness.

This additional .30 mm caused the substrate to exceed the maximum substrate width of 4.6 mm. The 30° break also caused a razor-like edge that was easily damaged during handling.

The laser power was increased in an effort to laser scribe the sapphire completely through its thickness of the 30° cleavage side. Although the laser was able to scribe through the sapphire, it appeared to leave an excessive amount of debris on the working surface of the substrate. An Auger (AES) analysis of that surface debris showed it to be Al and O. This deposit was sapphire that melted and redeposited on the surface near the scribe line. The deposited Al_2O_3 was impossible to remove without damaging the metallization pattern.

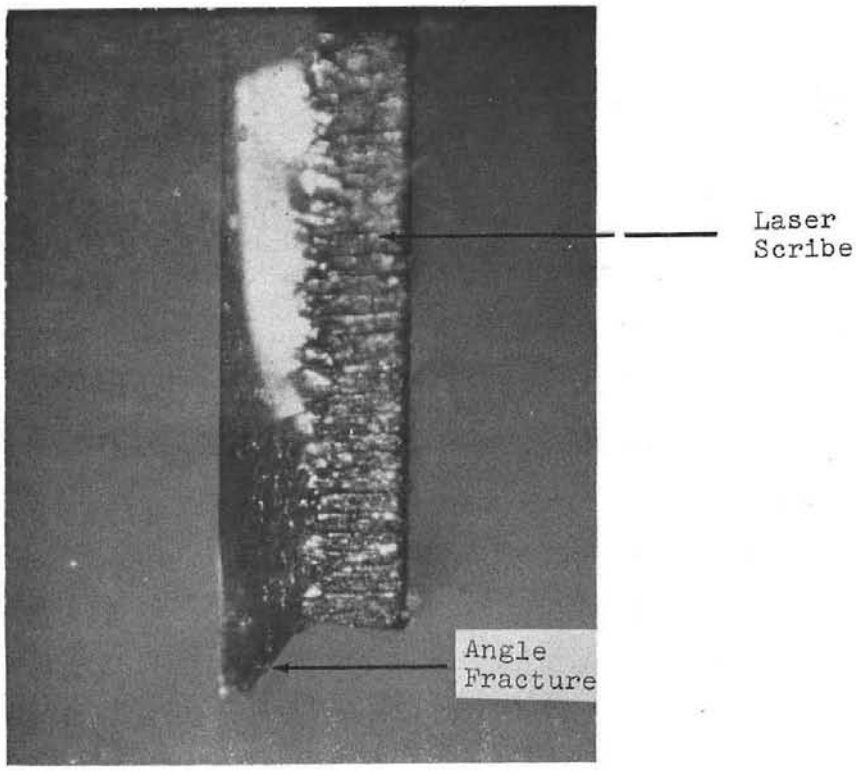


Figure 5. 30° Break Direction

Successful Sizing

In reviewing the crystal orientation of the sapphire, it was determined that the 30° break direction was caused by the tendency for the sapphire to break along a natural crystal cleavage plane. This plane was the r $(1\bar{1}02)$ plane and was actually 32° 26' off the predetermined metallized surfaces. The other two breaks were in the a $(21\bar{1}0)$ face and were at 90° to the working surface.

Sherry Spencer[7] suggested that the sapphire be pulled in a different orientation. Specifically, she recommended growing the sapphire parallel to the 4 plane so that scribing would actually make use of easy cleavage planes oriented approximately orthogonal to the r planes. Material pulled in this direction was ordered, and the laser was used to scribe this new material. The laser settings were: table speed .254 mm/sec, 40 to 50 watts with rep. rate of 15 KHz. After scribing and breaking, the edges were examined with the scanning electron microscope. The break was accomplished perpendicular to the working surface on all four sides (Figure 6).

Conclusions

One possible alternative to laser scribing and breaking would be to size the substrate larger than needed, then grind the edges until the desired size is reached. This was apparently the method used by the sapphire vendors to get the original 25.4 mm x 25.4 mm substrates. There are problems in fixturing for this operation to assure right angle lapping, and there would be special handling required to avoid damage to the fine line multilayer metallization patterns on the substrates.

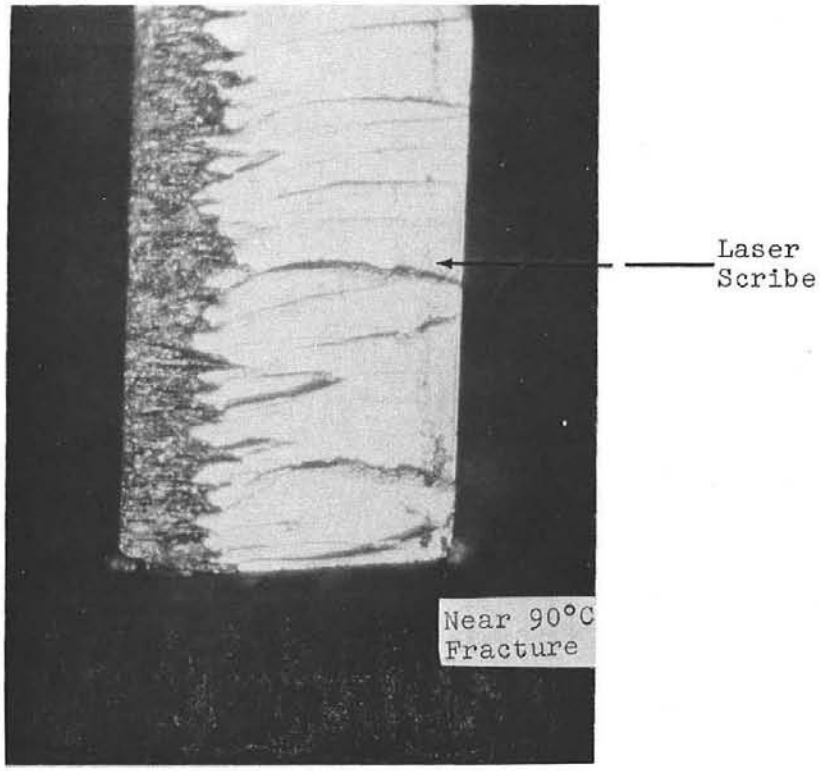


Figure 6. Scribe and Break of Sapphire Grown Parallel to the r Plane

If dimensional tolerances are critical, sizing of single crystal sapphire substrates can be a problem. Sizing attempts by using grinding wheels and wire saws and laser scribers proved inadequate for single crystal sapphire. The cleanest edge resulted from laser scribing. However satisfactory sizing was not accomplished until the sapphire was supplied with the orientation of the r face perpendicular to the scribe beam. Care must be taken to set the parameters of the laser in such a way to avoid the deposit of molten sapphire on the surface during scribing. The laser must also be carefully focused to overcome the near transparent appearance of the sapphire to the laser beam. With these factors taken into account the referenced[5] laser scriber was used to successfully size single crystal sapphire up to 0.38 mm in thickness.

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