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CHARACTERIZATION OF CERAMIC SUBSTRATES

By A. Laudel

Published February 1979

Final Report

Prepared for the United States Department of Energy
Under Contract Number DE-AC04-76-DP00613.

**Kansas City
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By A. Laudel

Published February 1979

Final Report
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CHARACTERIZATION OF CERAMIC SUBSTRATES

BDX-613-2057 (Rev.), Final Report, Published February 1979

Prepared by A. Laudel

Characterization techniques were developed to improve receiving inspection and in-process testing of 99.5 percent alumina substrates used for manufacturing hybrid microcircuits. Many of these tests have been implemented into production; other tests have been recommended for further development. The tests developed were scanning electron microscope (SEM) measurement of grain size; low angle light inspection for surface defects; non-contact thickness and camber test; dye penetrant check for porosity and cracks; and SEM inspection of surface finish in holes. The effect of raindrop (variation of light transmission through substrates) and polished high spot defects were determined.

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SUMMARY

The purpose of this project was to develop characterization techniques for ceramic substrates used in hybrid microcircuit (HMC) manufacturing so that scrap resulting from substrate defects could be reduced. Scrap is produced in subsequent HMC production operations when substrates have defects, such as pits, scratches, burrs, pinholes, bumps, dents, waviness, porosity, and hazy appearance. These defects result in the following types of problems: breakage during lead frame bonding; failure to break straight after laser scribing; high sheet resistivity in certain areas; lowered adhesion in certain areas; voids in resistor and conductor areas; high resistance vias (metallized holes in the substrates to connect frontside network to backside ground plane); and thin-film network (TFN) breakage caused by cracks. This project consisted of a number of investigations into techniques for detecting, evaluating, and eliminating substrate defects.

A light inspection device was developed for inspecting pits, burrs, scratches, indentations, waviness, bumps, dents, haze, polished high spots, and pinholes. Inspection equipment based on this design is presently being used to inspect for haze and for uniformity of light transmission.

Grain size measurement techniques were studied. As a result of this study, an improved method using SEM photographs is being used for substrate lot acceptance. This method, in addition to being more accurate, gives a permanent record and shows typical grain structure for each lot of substrates.

An improved method to determine substrate density was developed to replace the sink-float (in a toxic liquid) test. Weighing substrate samples in air, then in water, and calculating the density has been adopted. With this method, a record of actual density rather than a go-no go indication is maintained, and the use of the toxic liquid is eliminated.

The laser reflectometer previously was used as an acceptance criterion for substrates. Studies during this project revealed that laser reflectivity had no correlation with grain size (the original reason for its implementation), and this method has been eliminated as an acceptance criterion. Laser reflectivity does provide information on trends in substrate quality.

An SEM method for determining via quality was developed and gives excellent detail of internal hole surfaces. This technique was implemented as the receiving inspection criterion for acceptance of green drilled holes.

Two lots of vendor substrates had a surface condition that resulted in gray-colored spots appearing on the substrate after cleaning. These spots had no adverse effect on performance; however, the spots were similar enough to the tantalum resistor in appearance that some substrates were rejected because the spots appeared to be shorts. These spots were caused by surface porosity. The most effective test turned out to be cleaning the substrates and observing whether they spotted. The vendor corrected the problem with this cleaning process and no further substrate spotting has been encountered.

A dye penetrant test was developed to accept or reject cracks around vias produced by ultrasonic drilling. This test has been implemented by Inspection and has proven effective in minimizing the number of high resistance vias encountered in production.

A contact type flatness-contour-thickness tester was built using two opposing probes with accurate position gaging. This tester provided information about typical substrate contours and thickness variations. In addition, it pointed out the necessity of continuing to make such measurements, but doing so with a non-contact type tester. A non-contact tester has been procured and will be characterized on a future project.

The continuous process sputtering machine becomes jammed if substrates break during the mechanical handling of the substrates in the machine. Weak substrates, such as those with cracks, must be eliminated before they are run through this machine. One manufacturer uses a drop test to determine mechanical integrity of substrates. An improved test was developed consisting of a pressurized diaphragm. This test provides a stress on the substrate: a good substrate will pass; and a bad substrate will break. Evaluation of this test to screen substrates before sputtering will be conducted on a future project.

DISCUSSION

SCOPE AND PURPOSE

The purpose of this project was to develop characterization techniques for ceramic substrates used in HMC manufacturing so that scrap caused by substrate defects could be reduced.

The scope of this project included development of the following tests.

- Develop low angle and transmitted light tests and corresponding acceptance criteria for screening substrates to minimize defects and contaminants which can have a detrimental effect on thin film properties in subsequent processing.
- Develop a satisfactory grain size test, correlate grain size with laser reflectivity, and determine the usefulness of laser reflectivity testing.
- Investigate dielectric constant tests and determine the appropriate test for substrate acceptance testing.
- Determine if the in-house compositional analysis capability is adequate to monitor vendor trends in substrate composition.
- Develop tests for screening surface porosity and spotting.
- Develop an improved test for substrate density.
- Investigate appropriate tests and procedures for determining camber, flatness, and thickness profiling of substrates.
- Develop inspection techniques for vias (holes drilled in ceramic which when metallized, become conductive paths from frontside to backside metallization).
- Investigate substrate strength, fracture toughness, etc., and make recommendations as to implementation of tests.

PRIOR WORK

Substrate manufacturers frequently make changes in substrate composition and processing. It is necessary that the trends of density, composition, and other properties be monitored so that changes in vendor processing are observed, and their consequences assessed. One vendor, questioned about a change in laser reflectivity, said that over the past 3 years a continuing change in composition had resulted in higher density and lower surface porosity.

Thin-film networks for certain HMCs require better control of dielectric constant, thickness, via location, finish of interior via walls, and many other parameters than are standard in the industry. Specifications and testing techniques are required to ensure these requirements are being met.

Some substrates have demonstrated a surface porosity which resulted in spotting of the substrates after cleaning. These spots were so similar in appearance to the resistor film that they interfered with visual inspection of HMC networks after photolithography. It is necessary to have the capability of assuring that substrates will not spot before acceptance.

Experience in procuring and using substrates indicated the following improvements in substrate inspection techniques were required.

- Improved inspection techniques for assessing grain size, surface defects, density, and cracks;
- Tests to ensure special control of camber, thickness, via location, and internal surface finish of vias;
- Techniques to eliminate the spotting problems; and
- Techniques to accumulate information concerning trends in substrate fabrication so that problems can be anticipated. This involves investigation of density, composition, porosity, and laser reflectivity.

ACTIVITY

Surface Defects

Investigation of various illumination techniques at Bendix Kansas City and discussions with in-house and vendor personnel who visually inspect substrates indicated that very low-angle illumination was the best approach for enhancing visibility of surface defects. Design sketches for a low angle illumination fixture were obtained from a vendor and a fixture was built and evaluated.

Figure 1 shows the illuminator and Figure 2 shows two substrates being illuminated by the low angle light. The illuminator projects and collimates the light into a narrow beam. The substrate is oriented so that the beam crosses it at a shallow angle. Also, the center portion of the holder has been cut away to provide for illuminating through the substrate to inspect for pinholes.

Figure 1. Low Angle Light Illuminator

Figure 2. Substrates Under Low Angle Light

A number of types of defects were found. In some cases, the source of defects is known and the adverse consequences on subsequent processing are evident. These concepts are discussed in Appendix A.

A sample of 923 substrates from two manufacturers was observed by inspection personnel to determine the frequency of occurrence of various types of defects. Table 1 lists these results.

The line scrap after laser trimming of resistors and breakage during lead frame bonding were reviewed for 1030 thin-film networks. The reason for scrapping the substrates was noted and the percent occurrence of that reason for scrapping was also noted.

Using the information from Tables 1 and 2, the resulting scrap rate can be projected. Foreign material and dents generally do not contribute to scrap, but the area of polished high spots contributes to the probability of rejects. Substrates with foreign material and dents and substrates with less than three polished high spots are not considered rejects. Therefore, referring to Tables 1 and 2, if 8.2 percent of the substrates are rejected, approximately 4 percent of the thin-film networks which are processed would no longer be scrapped.

Grain Size

A metallurgical microscope was formerly used to determine the size of aluminum oxide grains which make up the substrates. A reticule was placed in the eye piece which overlayed a circle on the image of the substrate. Magnifications were in the range of 500 to 2000X. The number of grain boundaries intersected by the reticule circle were counted and the grain size was calculated.

This process is very difficult because there is very little optical contrast between grains and grain boundaries since the substrate is white and translucent. Figure 3 shows microphotographs at 500 and 2000X through the metallurgical microscope. The photographs themselves are not useful in determining grain size. However, a skilled operator can adjust light angle, focal depth, and contrast during the measurement and get a relatively accurate reading. There is, however, error introduced because of the lack of resolution.

As part of this project, a technique using a scanning electron microscope was evaluated and adopted. It is the same method which is used generally throughout the ceramic industry and is in accordance with ASTM grain size determination techniques.¹ Appendix B outlines the grain size determination used by Bendix and Figure 4 shows the typical SEM photographs which are used in determining the grain size.

Table 1. Substrate Defect Occurrence, Sample 923 A and C Substrates

Type of Defect	Percent of Defects	Rejection Level* (Percent)
Polished High Spots	9.0	5.0
Foreign Material	3.0	--
Dents	2.5	--
Pinholes	2.0	2.0
Impressed Patterns	0.4	0.4
Scratch	0.3	0.3
Ripples	0.3	0.3
Haze	0.2	0.2
Totals	17.7	8.2

*This is the expected rejection percentage using the revised criterion.

Table 2. Processing Line Scrap, Sample 1030 TFNs

Reason for Scrap	Percent of Sample	Percent of Processed TFNs Possibly Saved With Proposed Substrate Scanning
Broken After Scribing	1.5	1.0
Resistor Over-High-Limit	7.3	1.0
Ceramic Crack	0.1	--
Scratch	0.2	0.1
Voids in Resistors or Conductors	7.2	1.0
Broken At Lead Frame Bonding	1.5	1.0
Totals	17.8	4.1

500X

2000X

Figure 3. Grain Structure of HMC Ceramic Substrates
as Viewed Through a Metallurgical Micro-
scope

AVERAGE SIZE 1.9 μ m
3000X
CHO66995
LOT NUMBER D-20-5
CONTROL NUMBER 023319

Figure 4. Typical SEM Photographs Used for Grain Size Determination

Laser Reflectivity Versus Grain Size

The laser reflectivity number is obtained for each lot of substrates received at Bendix. The laser reflectivity test was introduced in the first place as having a relationship with grain size. It was observed in the testing that there was up to 10 times the change in laser reflectivity number with a very small change in the grain size.

This activity was to correlate laser reflectivity versus grain size to determine if there truly was a relationship. The laser reflectance of 25 substrates from each of two vendors were measured; then grain size was determined using the circle intercept method. The data obtained from this experiment is plotted in Figures 5 and 6. There is no substantial correlation between laser reflectivity readings and grain size.

Since there is no measurable substrate parameter that correlates with laser reflectivity, laser reflectivity has been eliminated as an acceptance test; it is being used to monitor trends in vendor process changes.

Dielectric Constant

Experiments were conducted to determine the capability for making repeatable and reliable measurements of dielectric constant of 99.5 percent alumina substrates. Measurements were made using a test cell and a modified version of the two fluid method described in ASTM D-150, paragraph 7.2.3. The repeatability of measurements is approximately ± 4 percent.

The method uses a silicone fluid which is highly sensitive to relative humidity. Measurements must be made in a shielded room. A sensitive capacitance bridge is used and a 24-hour warm up time is recommended. The method is very sensitive to temperature. The top frequency is limited to 1 MHz. If care is not taken, bubbles will form in the silicone fluid, and the bubbles will affect the reading. The thickness of the substrate must be accurately known.

The only way in which this method of measurement would be useful would be to make a measurement on a standard sapphire substrate, then immediately on the unknown substrate, and to have the readings corrected as required. Using this technique, repeatability of ± 0.5 percent might be anticipated.

A new method uses measurements at the desired frequency and claims repeatability to ± 0.1 percent of the dielectric constant relative to a known standard substrate.² Bendix product engineering studied this method and it is currently being used with a repeatability of ± 0.03 percent. Substrate thickness changes of ± 25 μm reflect a ± 0.18 percent change in apparent dielectric constant.

Figure 5. Laser Reflectivity Versus Grain Size, Vendor A

Figure 6. Laser Reflectivity Versus Grain Size, Vendor C

Compositional Analyses

Compositional analyses were performed on several substrates by the Bendix Test Laboratory using emission spectroscopy. The procedures were reviewed; it was found that compositional analyses could be made repeatably and that the Bendix analyses were in essential agreement with the manufacturer's published data.

All substrates were found to be within the required tolerances. Actual compositional analyses which were run are found in Table 3. It will be noted that in all cases the actual composition of aluminum oxide exceeds the 99 percent minimum which is required. The largest impurity is SiO_2 . This is an intentional additive to give the ceramic the desired properties. The magnesium and calcium are also in the form of oxides and have been added to the ball mill batch as grain growth moderators. The rest of the elements present are impurities which vary greatly from lot-to-lot of raw aluminum oxide furnished to the substrate vendors. These impurities do not seem to have a substantial effect on the substrate properties.

Porosity Tests and Substrate Spotting

Gray spots have appeared on some substrates after they have been processed through cleaning on two lots of substrates purchased from the same vendor. The spots cannot readily be discerned from the sputtered tantalum nitride on thin-film networks. The similarity in color between the spots and the sputtered tantalum has resulted in scrapping otherwise good thin-film networks because they appear to be shorted. Substrates which exhibit spotting have a lower physical density. The vendor of the substrates, as well as other independent sources, have indicated that the most probable cause of the spots was surface porosity.

An attempt was made to develop a test which would predict the tendency to spot through observation of the surface porosity. The test must be less complicated to run and less expensive than to clean a batch of substrates and look at them for spotting.

The test which came closest to meeting this objective involved applying a water soluble fluorescent dye penetrant to the substrate, rinsing the excess penetrant away with water, drying the substrate, and viewing it under ultraviolet light. The fluorescent dye penetrant test is very sensitive in showing cracks, but it is not conclusive in predicting spotting.

The use of dye penetrant as an inspection technique to anticipate spotting was determined as follows.

Table 3. Composition of Analysis Summary by Emission Spectroscopy

Element	Sample (Percent of specified element)				Vendor
	1	2	3	4	
Al ₂ O ₃	99.26	99.36	99.60	99.55	M
Si	0.48	0.38	0.20	0.26	
Mg	0.16	0.11	0.14	0.16	
Ca	0.024	0.084	0.02	0.045	
Fe	0.043	0.036	0.01	0.023	
Mn	0.002	0.0036	0.0016	0.002	
Sn	0.001	0.001	0.001	*	
Ga	0.021	0.022	0.008	0.0005	
Ti	0.01	0.009	0.011	0.004	
K	*	*	*	0.004	
Cu	*	*	*	0.002	
Ag	*	*	*	0.001	
Al ₂ O ₃	99.19	99.23	99.09	99.17	C
Si	0.45	0.40	0.49	0.49	
Mg	0.21	0.18	0.20	0.17	
Ca	0.088	0.098	0.13	0.11	
Fe	0.033	0.031	0.047	0.040	
Mn	0.003	0.002	0.003	0.002	
Sn	0.001	*	*	*	
Ga	0.013	0.032	0.02	*	
Ti	0.007	0.025	0.019	0.015	
K	*	*	*	*	
Cu	*	*	*	*	
Ag	*	*	*	*	
Al ₂ O ₃	99.17	99.25			A
Si	0.32	0.24			
Mg	0.28	0.45			
Ca	0.076	0.05			
Fe	0.036	0.03			
Mn	0.0027	0.002			
Sn	0.002	*			
Ga	0.021	0.005			
Ti	0.092	0.004			
K	*	0.004			
Cu	*	0.002			
Ag	*	0.001			

*Not detected.

- Four sets of substrate samples were selected. These sets were designated A, A-spotting, M, and C.
- Dye penetrant was applied to all substrates for two minutes, then rinsed off.
- Photographs of the fluorescent areas of the substrates were taken with ultraviolet light in a light-tight box.
- The substrates were then cleaned and examined for spotting.

Only the A-spotting type showed spots. The spots in most cases coincided with areas of dye penetration, but did not occur in all areas of dye penetration. They were, however, generally in the areas with the most intense fluorescence.

Figure 7 shows a typical comparison of the four types. The intensity of fluorescence depends upon the rinsing technique, the temperature, etc., and will vary by a factor of about two. Some of the type C substrates were brighter under ultraviolet light than the type A-spotting substrates. Thus, the test is not selective enough to use as a receiving test.

The vendor of A substrates has modified the process by increasing substrate density. Therefore, for the time being it is not necessary to test for substrate spotting since it will become evident in cleaning.

The following methods of measuring porosity were considered; however, they were never implemented.

Mercury Intrusion Porosimetry:	More related to semi-closed bulk porosity than to open surface porosity.
BET Surface Area Measurement:	Uses desorption of gas from surfaces by firing in a vacuum. In order to have enough gas for analysis, many substrates would be required. The reading would be an average of the group and the sensitivity ± 20 percent.
Beta Backscatter:	Five measurements were made on each of four sample substrates using a carbon (150 kV) source. No correlation with porosity was made.

A

SPOTTING A

M

C

Figure 7. Substrates With Dye Penetration Before Cleaning

Fuchsine Dye:

No dye penetration was visible.

Substrate Density Versus Substrate Spotting

As mentioned in the preceding section, a problem was encountered on two lots of substrates on which gray spots of various sizes appeared on the working face of some, but not all, of the substrates after they were cleaned. These spots did not affect sheet resistivity, thermal coefficient of resistance (TCR), life stability of resistors, or thermocompression bondability. However, the color of the spots was so similar to that of the sputtered tantalum-nitride film that many thin-film networks were scrapped because spots appeared to be shorts between legs of resistors or between conductors.

Physical densities of individual substrates from these two lots ranged from 3.79 to 3.87 g/cc compared to the requirement of 3.80 minimum. The Bendix Receiving Inspection test was a go, no-go flotation of 22 substrates in thallium malonate formate solution having a density of 3.79. There were no failures in the Receiving Inspection test for these two lots. Substrates which spotted after cleaning had a density range of 3.79 to 3.84, while densities of substrates which did not spot ranged from 3.82 to 3.87 (weigh-in-air/weigh-in-water measurement).

Some large spotted areas were broken away from substrates, and their densities were measured. The densities of the most spotted areas ranged from 3.78 to 3.82, while unspotted pieces of the same substrates ranged from 3.82 to 3.88. Thus, spotted areas have a lower average density than unspotted areas but density cannot be used as a selection criterion to anticipate which substrates will have spots.

The ceramic vendor has changed the composition of the ball mill batches of ceramic since the time of the spotting problem. This change has increased density and decreased the porosity of the substrates.

Weight in Air - Weight in Water Method of Substrate Density Measurements

The substrate densities which are quoted in manufacturers specifications and in literature are typically found by weighing the substrate sample in air, then weighing it in water, and calculating the density using the following formula:

$$\text{Density} = \frac{\text{weight (air)}}{\text{weight (air)} - \text{weight (liquid)}} \times \text{density (liquid)}.$$

In working with this test, it was noted that there are several potential sources of error: bubbles adhering to the ceramic or the clip on which it is suspended, water temperature, impurities in the water, and substrate cleanliness. The temperature and purity variables can be minimized by using deionized water which has been allowed to sit for at least an hour so that the test can be run at room temperature.

A question was raised as to whether greasy fingerprints would fill in surface porosity, prevent wetting, and introduce an error in density measurements. An experiment was run to determine the effect of substrate cleanliness on the measured density. Substrates were handled with bare hands ranging from no fingerprints to a complete coverage of fingerprints. Density was then measured. Substrates were cleaned using the standard cleaning process (ultrasonic cleaning in trichloroethylene, acetone, alcohol, and a wetting agent; followed by rinsing in flowing deionized water and baking at 900°C in a belt furnace). The density was again measured and the results are shown in Table 4.

Comparing the "Clean" and "Dirty" density measurements for each sample shows that substrate handling does not create a problem in making accurate density measurements on substrates.

Formation of bubbles can be minimized by dipping the substrate in a solution containing a wetting agent, then immediately transferring it into the weight-in-water measurement. An experiment was run to check whether dipping substrates in a wetting agent solution would give a different density measurement than when no wetting agent was used. The wetting agent substantially reduced the tendency for bubbles to adhere to the substrate without materially affecting the density measurements.

The density measurement method, which is in accordance with ASTM D-792, has been adopted. Details of this method are found in Appendix C.

Thickness and Flatness Tester

A method for measuring the thickness and flatness of substrates using conventional LVDTs (linear variable differential transformers) as transducers was developed. The LVDT is a spring-loaded cell which presses a probe tip against the item to be measured and produces an electrical signal that is linearly proportional to position. By varying the excitation to the LVDT, a zero correction can be made electrically without changing the position of the LVDT.

The system shown in Figure 8 was assembled for measuring both thickness and flatness. The key elements of this system are the granite surface plate and two opposed LVDTs which have sapphire

Table 4. Effect of Cleanliness on Density

Sample Number	"Dirty" Substrate Density	"Clean" Substrate Density	Number of Fingerprints
1	3.81	3.81	None
2	3.84	3.84	1
3	3.87	3.85	2
4	3.83	3.84	3
5	3.88	3.87	Completely covered
Average	3.846	3.842	

gaging heads. One of the LVDTs is nested inside the granite plate such that the gaging tip is flush with the surface. The other gaging head is mounted on a standard height gage.

Since LVDTs require contact of the surface to be gaged, a study was conducted to determine if the gaging tips or the substrates would be damaged during measurement. In the study, several gaging heads were used with various gaging forces. Steel probes with tip radii of 2.3 mm, 4.1 mm, and 51 mm were studied. A sapphire tip with a 51 mm radius was also used. The substrates were damaged by traversing a steel tip across the same path 20 times. The amount of damage to the substrates was found to increase as the tip radius decreased and as the contact force increased. A 2.3-mm-radius tip was traversed across a substrate at 15 g of pressure. There was a significant flattened abraded area at the point of contact after 50 cm travel across the substrate.

The sapphire tip was traversed across the substrate at pressures of 15, 50, and 100 g. No damage to the substrate or probe tip was evident. However, it is expected that a polishing effect will occur and that the substrate must have some type of protection from contact with the tip and with the granite block.

The gaging system was calibrated using gage blocks. The repeatability of the system for thickness measurements was $\pm 3.8 \mu\text{m}$ at a 2 sigma confidence level. This repeatability includes errors caused by misalignment of the probe tips and inabilities of the operator to measure thickness at precisely the same location.

Figure 8. LVDT Setup

The repeatability of the system for flatness measure was $\pm 6.1 \mu\text{m}$ at a 2 sigma confidence level. An additional error was present when making flatness measurements because the probe force tends to lift the substrate above the reference surface.

Foreign material imbedded in the surface plate caused gaging errors and substrate damage. Since it is essential to maintain clean surfaces, a system was developed in which contact of the probes and the granite block to the substrate was eliminated by adding clean room paper to the top and bottom of the substrate. The paper remains in contact with the substrate and the probes and the granite block are in contact with the paper. An additional loss of resolution of approximately $\pm 6 \mu\text{m}$ comes about from the lack of uniformity of thickness of the paper.

This system is satisfactory for destructive sample testing, but not as a routine non-destructive test. It was used in flatness

testing of substrates to determine the improvement in flatness which is obtained after refiring the substrates. For accurate nondestructive flatness testing a non-contact system is needed.

Flatness Test

Vacuum Hold Down Type

One of the substrate vendors uses a 100 percent screening test for flatness of substrates which consists of measuring the vacuum when a substrate is placed on a vacuum hold down fixture. If the substrate is flat and seals to all surfaces, a high vacuum is obtained. When a substrate is not flat, a lower reading of vacuum is obtained because of the leakage.

Design sketches were obtained from the vendor and a tester was built (Figure 9). Plexiglas was used in the area of contact with the substrate to minimize substrate contamination.

Most substrates are slightly convex such that when they are placed on a tester backside down, a relatively high vacuum is achieved. Therefore, tests must be made working face down to obtain maximum selectivity.

A number of substrates were tested using the experimental fixture, and the vacuum measured was correlated with camber as determined by the LVDT tester in a camber mode. It was found that there was not a sufficient relationship of the vacuum reading to the flatness to use this method for screening or for receiving inspection.

It was found that the vacuum hold down technique was much more sensitive to ripples near edges and prominent raised places than to the camber which is defined by an adjustable parallel plate tester. Another factor introducing error is that substrates with a large camber, which is represented by a relatively uniform curvature, will be flattened by the vacuum hold down and will be considered acceptable.

Parallel Plate Tester

Flatness testing can be most easily and efficiently accomplished by the method that most manufacturers use. This is the parallel plate tester. The parallel plate tester is a plane of Plexiglas tilted at approximately 45° with a parallel plate of Plexiglas spaced away from it to allow substrates with acceptable camber plus thickness to slide through. If the substrates do not slide through readily, camber is above the limits set by the tester. It will be substantially less expensive to build a series of parallel plate testers at increments of $25\text{ }\mu\text{m}$ for studying camber distribution and for selecting substrates with acceptable camber for special requirements.

Figure 9. Vacuum Hold-Down Setup

A parallel plate tester was built and is being used for screening substrates after re-fire at 914 μm (0.036 in.) spacing. Acceptance level in receiving inspection is 991 μm (0.039 in.).

Non-Contact Gaging

A number of substrates were measured to determine the typical distribution that might be expected for substrate thickness using contact type gaging. Some substrates are relatively uniform in thickness, while others have a thickness range from side to side or top to bottom of as much as 37 μm . For RF applications, the most uniform, homogeneous thickness that is available is needed. Standard thickness tolerances for substrates are $\pm 50 \mu\text{m}$ and the best thickness tolerance available at a premium price is $\pm 25 \mu\text{m}$. Tolerance desired for the RF substrates is even better than this. Therefore, a screening technique which is able to select substrates where no part of the substrate deviates more than $\pm 6 \mu\text{m}$ from the required value is needed. It is also desirable to have the thickness testing be on a non-contact basis so that handling damage to the substrates is minimized.

A non-contact thickness gaging system was procured. It consists of a pair of capacitance probes operating in the same way as the LVDT probes were. Readout is displayed digitally as deviation from the nominal or in actual thickness with $\pm 0.25 \mu\text{m}$ resolution. Calibration is not necessary, since the nominal thickness is set up using a reference substrate.

This project was completed before the instrument could be evaluated and fixtures could be made for efficient handling of the substrates. A new project is being planned which includes completion of this fixturing and verification of the performance claimed by the manufacturer. Techniques for screening of substrates for RF HMCs will be developed, and these techniques will be incorporated in the production system and the instrument be transferred to inspection.

Via Inspection

Holes drilled through substrates and subsequently vacuum metallized to provide interconnections between the front side of substrates and the backsides are called vias. In development work on substrates, high resistance vias (over 10 $\text{m}\Omega$) with 6 μm gold thickness were encountered. The high resistance was traced to roughness in the hole wall caused by the hole fabrication method. Via holes range in diameter from 0.69 to 0.91 mm. The thickness of the substrate is approximately the same as the hole diameter. The surface finish inside the holes is very difficult to evaluate because the substrate is a translucent white and there is almost no optical contrast of even substantially defective areas. The

defects can be much more readily seen after metallization. Several techniques were evaluated to determine or inspect surface finish in holes. These are: scanning electron microscope; fisheye microscope; scribe, break and observe with conventional microscope; and conventional binocular microscope at relatively low magnification with backlighting through the hole.

Scanning Electron Microscope

The scanning electron microscope provided the best indication of surface finish within the hole. Use of the scanning electron microscope requires that a small piece of a substrate be used, and that the surface of the substrate be metallized to allow dissipation of the charge built up. Excellent detail is provided when the hole is sectioned, but satisfactory detail is obtained when viewing the hole from 45° without sectioning. Figure 10 shows typical scanning electron microscope photographs. The scanning electron microscope observation of hole finish is a destructive test. Even so, it has been recommended and adopted as the receiving inspection technique for green drilled holes, and in laboratory analysis of ultrasonic drilled holes and laser drilled holes.

Fisheye Microscope

Inspection of via walls can be made throughout the length of a via with a fisheye microscope. A fisheye microscope is a monocular microscope with a fisheye objective lens. The examination platform is opaque glass with a hole approximately the diameter of a via and is backlit. The interior surface of the via can be scanned by changing the focus of the microscope. A typical fisheye microscope is shown in Figure 11.

The contrast which is obtained using a fisheye microscope is poor because of the white translucent substrate material. Satisfactory contrast can be obtained after metallization of the via. It is possible to inspect uncoated substrates using the fisheye microscope, but considerable skill is required by the operator and the judgements are subjective. It is not a suitable method to place in receiving inspection.

The fisheye microscope is used extensively in the printed circuit board industry to inspect plated-through holes. However, plated-through printed circuit board hole diameters are considerably larger than the vias which are of interest, and the surface is metallized. It is a nondestructive test, and techniques for enhancing the contrast would be a profitable subject for further investigation.

Figure 10. Hole Inspection Microscope

Figure 11. Holes as Viewed Through Hole Inspection Microscope

Scribe, Break, and Use of Low Angle Lighting With Conventional Microscopy

This method of inspection involves scribing a substrate through the center of the via, breaking the substrate at the scribe line, and then visually examining the via using a microscope while the side-lighting is moved around to accomplish the optimum contrast. It is possible to examine unmetallized holes with this method. Examination must be performed using relatively low magnification in order to have a sufficient depth of field to observe the defect. Viewing should be performed shining the light first from one side, then from the other in order to see all of the defects. This method requires some skill, is a destructive test, and does not provide a record since it consists of a substantial number of successive observations with changing angle of light and reversal of direction of the light.

Conventional Microscopy

A conventional microscope is used to view a substrate on an opaque stage. A small hole in the center of the stage and axial backlighting are required. The microscope viewing is at 10 to 45° away from the axis of the hole. All sides of the hole can be viewed by rotating the substrate.

The best configuration would be to make a rotary microscope stage that can be tilted from 10 to 45° with a hole in the center and a vacuum hold down. The light is mounted to the stage support. The hole must be accurately centered so that rotating the stage does not change the focus of the microscope on the hole.

Substrate Strength

The purpose of this investigation was to determine the homogeneity of the substrates with respect to strength and modulus of elasticity, the extent to which localized stress around holes can be determined, and the feasibility of developing a non-destructive screening test that will reject or break substrates which have cracks.

A series of 14 holographic interferograms, summarized in Table 5, were run on substrates from each of three manufacturers. The holograms give a rough estimate of the strain and the modulus of elasticity for each part. The interferograms were made with a pressure differential of 0.48 Pa above a preset pressure of 12 Pa. The samples were supported around their perimeter and uniformly loaded on the back surface with air pressure. Each white fringe in the interferogram represents an increment of approximately 250 nm displacement toward the observer, counting from zero displacement at the edges of the substrate. Knowing the strain which is indicated by the interferogram, the stress may be calculated.

Table 5. Identification of Holographic Interferograms

Sequence Number	Supplier	Remarks
10	A	Solid
11	B	Perforated
12	B	Perforated
13	C	Solid
14	Polished A	Solid
15	B	Solid
16	B	Perforated
17	B	Perforated
18	A	Rerun of Sequence Number 10
19	A	Solid
20	A	Solid
21	A	Solid
22	A	Solid
23	A	Solid

It was noted that one sample (sequence 10) showed nearly twice as much deflection for the same pressure differential as other samples. Figure 12 shows the original interferogram and a rerun and compares them with an interferogram from a more typical sample. It might be implied that the low modulus of elasticity would result in low strength. This substrate was tested for dye penetration and was found to have virtually none. It was tested for density; 3.82 which is normal. The laser specular reflectivity was within a normal range for the substrate. Therefore, this substrate, along with two other substrates from the same vendor, were pressurized in the same mode (supported from edges) and the air pressure behind the substrates was increased until they broke. The results from this test are shown in Table 6. The ultimate stress of 596 MPa is higher than the typical reading, and may be related to some simplifications in the calculation. Typical strengths for these substrates are 350 MPa. In addition, one other substrate broke at a low applied pressure 76 kPa where typical values on breaking hundreds of substrates have good substrates breaking between 90 and 124 kPa. Even though this is a small sample, it is evident that modulus of elasticity does not predict the ultimate strength and that the most significant information would be obtained from strength testing.

**SEQUENCE 10
LOW MODULUS OF ELASTICITY**

**SEQUENCE 18
RERUN OF SEQUENCE 10**

**SEQUENCE 20
NORMAL MODULUS OF ELASTICITY**

**SEQUENCE 16
TYPICAL FROM VENDOR B**

**SEQUENCE 13
TYPICAL FROM VENDOR C**

Figure 12. Interference Holograms

Figure 13 shows the interferogram for a substrate with holes. Deflection is substantially increased by the presence of the holes and subsequent testing has shown that strength is proportionately decreased. However, no localized stress around the holes is indicated, and the reduction in strength is caused as much by the reduced cross-sectional area as by localized stresses. Localized stresses will produce small deflections compared to the overall deflection; therefore, holographic interferometry is not inherently very sensitive to localized stress.

A series of substrate samples were sent to the test laboratory to determine typical flexural strength modulus of rupture and modulus of elasticity. Results from this study are shown in Table 7.

A study was also initiated to determine the mechanics of fracture of 99.5 percent alumina substrates. This survey was done using techniques refined by Evans and others.^{3,4,5} The fracture mechanics test employs a double torsion sample which is basically a flat plate with a groove down the middle to guide the crack. The plate is loaded in three-point fashion at one edge of the groove causing the crack to propagate down the groove. The load is monitored and the fracture toughness calculated using the load and the geometry of the sample. In this case, a laser scribe was used to make the groove to an average depth of about 75 nm or about one-ninth of the total thickness. Table 8 shows the results for samples from two vendors. The data obtained compared closely with that reported by Evans.⁴ An error analysis was run and it was determined that the test accuracy was ± 6.5 percent. Therefore, no significant difference in fracture toughness between substrates is indicated.

The principal limitation of this technique is the use of a laser scribe to make the grooves in the sample. Each of the laser pulses produces a crater and the scribe line is a series of craters. Looking down the crack, the sharp hills and valleys along the bottom of the scribe serve as crack arresters. Therefore, the fracture toughness values determined by this test are slightly higher than they should actually be.

Elimination of substrates which are weak or which are already cracked would be of great value. A test fixture was built which supported a substrate on a rubber base around its perimeter and which applied air pressure to the opposite side through a rubber diaphragm (similar to an innertube). A small chamber below the substrate provided space for fragments in case a substrate broke, and limited the expansion possible for the rubber diaphragm. A preliminary investigation of fracturing properties was run. Good substrates from three vendors all sustained a range of air pressure from 9.7 to 13.1 kPa before breaking. Several substrates were introduced into the tester in which dye penetrant indicated that cracks were present. These substrates all broke at below 6.9 kPa.

Table 6. Results of Ultimate Stress Test

Sample	Pressure (kPa)	Ultimate Stress (MPa)
1	103	596
2	103	578
3	76	440

Substrates with holes break at reduced strengths proportionate to the number of holes in them. Further work must be done to determine whether substrates with holes which also have cracks can be selectively eliminated.

A screening test using 6.9 kPa for elimination of weak substrates appears to be feasible. Further work is planned for streamlining the test to be used in production.

Dye Penetrant Inspection of Vias

A fluorescent dye penetrant test was developed on this project to find cracks in substrates. It has been demonstrated that cracks which are as narrow as 0.1 μm and become visible to the naked eye when illuminated with ultraviolet light. A light-tight box with a camera mounted on the top was built to study effectiveness of dye penetrant testing. In the box was a substrate holder and alternate light sources (ultraviolet and low angle white lights). Figure 14 shows some typical ultrasonic drilled defects which are not readily visible with white light illumination. Particularly, the figure shows a series of radial cracks and a network of cracks under the surface parallel to the surface over the entire area which shows up bright. The bright line perimeter is where the crack network approaches the surface but does not break through.

In the investigation, three fluorescent dye penetrants were considered. All three penetrants were equal in showing surface porosity and cracks. The Non-Destructive Testing group already was using one penetrant for other applications and had it set up as a standard. Therefore, it was chosen to be further evaluated.

Five substrates were dipped halfway into the dye penetrant, then the surplus penetrant was rinsed away. These substrates were then processed through cleaning, metallization, photolithography,

Figure 13. Interference Hologram,
Substrate With Holes

stabilization, and CAN etch. Lead frame bondability pull tests were run and bond strengths with 0.18 mm lead frames were 1.4 kg $\bar{X}$ for the uncoated side and 1.41 kg for the coated side. The failure mode for most leads in both tests was B type.

A 10 g sample of the dye penetrant was sent to the Test Laboratory for analysis. The volatiles were boiled off at 300°C, then the temperature was raised to 900°C to simulate the cleaning furnace. There was only 30 ppm of the original sample left as ash. This was analyzed by emission spectroscopy and the predominant constituents were found to be gallium oxide with traces of iron, nickel, and tin. It can be concluded that the use of dye penetrant does not adversely affect HMC bondability.

This dye penetrant test has been adopted in production to inspect for defective ultrasonic drilled vias.

Raindrops

A phenomenon has been encountered on substrates which have been sputtered with tantalum. It consists of areas having higher light transmission than the normal substrate and is known in the industry as "raindrops". The total pattern sometimes appears as a partial coverage of various size overlapping circles as shown in Figure 15. Sometimes the raindrops are centered around vias, more often than is statistically probable, while other vias are not affected.

Sheet resistivity in the areas of the raindrops is approximately 10 percent higher than sheet resistivity in other areas of the substrate.

When the tantalum is etched off the substrate, there is no enhanced light transmission in the area. It is evident that the raindrops are a surface phenomenon on the working face of the substrate, and that it is influenced by a very thin surface layer.

Table 7. Flexural Strength Properties of Ceramic Substrates

Sample Number	Width (mm)	Thickness (mm)	Load to Failure (N)	Flexural Strength Modulus of Rupture (MPa)	Modulus of Elasticity (GPa)
B					
1	2.545	0.71	31.6	374	361
2	2.545	0.71	32.0	379	350
3	2.543	0.69	31.6	403	394
4	2.545	0.69	31.1	396	396
5	2.550	0.71	36.9	436	371
6	2.545	0.69	29.8	379	386
7	2.545	0.69	32.5	414	370
8	2.545	0.69	34.7	442	378
9	2.545	0.69	32.9	419	399
A					
10	2.545	0.71	20.5	242	339
11	2.548	0.71	34.2	405	345
12	2.545	0.71	34.2	405	346
13	2.545	0.69	32.9	419	368
14	2.548	0.69	31.1	396	356
15	2.545	0.69	28.5	363	354
16	2.545	0.69	30.7	391	365
17	2.545	0.69	28.9	368	373
18	2.548	0.69	30.7	390	370
D					
19	2.545	0.69	28.5	363	378
20	2.545	0.69	40.5	515	383

The vendor of the ceramic has encountered raindrops on substrates in their plant. A temperature profile to remove raindrops was recommended which greatly resembles the Bendix re-fire cycle except that it is 1450°C for only two hours. It was theorized by the vendor that the raindrop formation occurred at approximately

Table 8. Fracture Toughness Test
Results on Alumina

Sample	Fracture Toughness (MPa $\sqrt{m}$)
(Vendor A)	
1	3.07
2	3.07
3	3.07
Average	3.07
(Vendor B)	
1	2.49
2	3.18
3	3.12
4	3.31
Average	3.10

1000°C and was related to a slow cooling rate at 1000°C. This 1000°C temperature is probably encountered from time to time in the belt furnace cleaning cycle.

The present incidence appears to range from 2 to 5 percent of the substrates for this vendor, and has produced as a consequence some over-high-limit (OHL) resistors. TCR and life stability were not affected on the sample which was investigated; therefore, the adverse consequences of raindrops appear to be limited to the increased reject rate for OHL resistors.

ACCOMPLISHMENTS

The following accomplishments were achieved as a result of this project.

- Light inspection techniques were investigated and a simple, effective tester was built. Testers based on this design are being used to inspect for haze and for raindrops.
- The technique for measuring grain size of alumina substrates was revised. Using the SEM, measurements are more accurate and there is a permanent record of the actual grain structure.

WHITE LIGHT
ILLUMINATION

ULTRAVIOLET ILLUMINATION
OF DYE PENETRANT

WHITE LIGHT
ILLUMINATION

ULTRAVIOLET ILLUMINATION
OF DYE PENETRANT

Figure 14. Defective Ultrasonic Drilled Holes

SHADED AREAS HAVE
NORMAL LIGHT TRANSMISSION

Figure 15. "Raindrop" Pattern

- The weigh-in-air/weigh-in-water method of density measurement was substituted for the sink-float in a toxic liquid method. The quantitative measurements which result give insight into quality trends of the manufacturer, and elimination of the toxic liquid contributes to safer operation of the plant.
- It was determined that there is almost no correlation between laser reflectivity and grain size; therefore, laser reflectivity was removed as an acceptance criterion and is being used only to follow manufacturing trends.
- A clear cut inspection method for vias was developed. SEM inspection of the internal hole surfaces gives excellent detail. This technique was implemented as receiving inspection criteria for acceptance of green drilled holes.
- A contact type flatness-contour-thickness tester was built using two linear variable differential transformers. Use of this tester provided insight as to substrate contours and

thickness variations. This work was sufficiently successful that a recommendation was made for a non-contact type tester for use by inspection.

- A flexible diaphragm type substrate strength tester was built and demonstrated non-destructive screening tests for substrate strength and for absence of major cracks.
- It was determined that the cause of spotting was surface porosity, that dye penetrant was a marginal test for surface porosity, and that the vendor who shipped two lots of spotting substrates had made process changes. No further spotting should be expected.
- A dye penetrant test was developed to accept or reject cracks around vias which were produced by ultrasonic drilling. This test was implemented in inspection.

FUTURE WORK

The following work is planned as a result of the development work done on this project.

- Implement 100 percent screening of incoming substrates with low angle and transmitted light for defects such as haze, pinholes, contour inflections, polished high spots, indentations, pits, burrs, scratches, and bumps. Prepare a book containing examples of acceptable and unacceptable defects. Train inspection personnel, revise the specification, and provide for disposition of the defective substrates.
- Develop a test to detect raindrops earlier in processing and investigate methods for removing or eliminating them.
- Characterize the noncontact tester. Prepare screening criteria to obtain better uniformity for RF substrates as required.
- Build a via inspection microscope and stage. Implement the use of this new tool.
- Run further experiments on using the diaphragm strength tester as a substrate screening tool. Consider the use of a bladder, pre-inflated to a required pressure, for speeding the operation. Implement use of this test to break unacceptably weak substrates rather than using them.
- Investigate the use of the diaphragm strength tester on drilled substrates.

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Appendix A

COMMON TYPES OF SUBSTRATE DEFECTS

Appendix A

COMMON TYPES OF SUBSTRATE DEFECTS

Polished High Spots

Polished high spots are areas of the substrate which reflect low angle light more readily than the surrounding material. In all cases, they occur on the higher portions of the front sides of substrates. They are caused by abrasion which the manufacturer uses to remove burrs and other ceramic debris from the front face of the substrate. This is typically done by scraping across the front of the substrate with another substrate. When the tantalum-nitride resistor film is sputtered over a polished high spot, the sheet resistivity after stabilization is about 25 percent higher than the surrounding resistive film. This scraping action also removes some of the glassy material which covers the surface of the substrate in the as-fired condition. When this silica is removed, the adhesion is lowered substantially. This contributes to bondability failures; while the higher sheet resistivity contributes to resistors being over-high-limit. Based on experimental verification using a monitor substrate, resistor thermal coefficient of resistivity (TCR), and life stability do not appear to be affected by the polished high spots.

Foreign Material

Foreign material is usually characterized by a piece of ceramic or a piece of packaging material which is loosely adherent to the substrate surface. Foreign material has not contributed substantially to rejects and subsequent processing since it appears to be removed adequately by the cleaning process. Stained areas, also, do not appear to cause rejects.

Pits and Dents

The adverse consequences of having pits and dents depends upon the contour of the defect. A steep-walled pit will cause a void in subsequent metallization. A dent produced from one side only has very little effect. A dent, which consists in a deflection of the entire substrate, can cause breakage of a thin-film network during lead frame bonding if the dent is in the area of the bonding. Dents are typically caused by fragments of refractory being on the "setter" blocks on which the ceramic is fired. Pits are usually caused by a fragment of ceramic dropping on the front face of the substrate, then being removed by abrasion.

Pinholes

Pinholes are caused by inclusion of gas producing material in the ball mill mix such that a bubble forms and blows out through the top surface of the substrate. Pinholes will frequently extend more than half way through the substrate. If a pinhole occurs on a resistor or conductor path, it can cause a void.

Impressed Patterns

In handling the green ceramic, grooves or other patterns will be impressed into the soft material. These patterns usually do not affect resistors or conductors, but do make an uneven surface onto which lead frames and beam lead devices are bonded. Some bonding failures can be traced to impressed patterns.

Scratches

When thin-film networks are inspected, one of the reasons for rejection is scratches across resistors. Scratches which are on the ceramic before the tantalum-nitride-sputtered film is deposited look the same as those which occur after deposition. Therefore, scratches on substrates contribute to rejects, even though there is nothing wrong with the films.

Ripples

Some substrates exhibit rippled surfaces either from nonhomogeneous mixing of the ball mill batch, nonuniform drying, nonuniform initial heating, or lack of flatness of the setter block. Ripples with 50 to 100 μm height and a length of 2.5 cm are sufficient to cause breakage of the thin-film network when lead frame bonding is performed. Ripples over 200 μm in peak-to-valley amplitude make it impossible to adequately focus the laser for scribing. If the laser is out of focus during scribing, substrates do not break along scribe lines.

Haze

Haze (finely divided particles which cannot be dusted off) is caused by firing alumina dust onto the surface of the substrate. Lead frames which are attached in hazy areas have reduced adhesion. Resistors in areas of haze have high sheet resistivity.

Burrs

Burrs are fragments of refractory or substrate material that are dropped on the surface of the substrate and are fired onto the surface. If burrs remain in place after cleaning, but are broken away after metallization, they leave voids in the metallization.

Curled Edges

All edges of the substrate being curled is caused by the die which produces the substrate blank being dull. The curvature at these edges is high enough to either cause substrate breakage during lead frame bonding or to bring the laser scriber out of focus so that substrates do not break in a straight line.

Appendix B

GRAIN SIZE DETERMINATION

Appendix 8

GRAIN SIZE DETERMINATION

The sample is composed of three substrates which are sent from receiving inspection.

A portion of each substrate is removed (broken off) for the SEM study.

One photograph is taken of each specimen at 3000X.

Each photograph is evaluated in three places using a standard 20 cm circumference overlay in accordance with ASTM E-112. The number of intercepts with this circle is counted.

The grain size is determined using the following formula.

$$GS = \frac{L}{(G-1)M}$$

where

L = 0.2 m,

G = the number of grain boundaries intercepted,

M = magnification, and

GS = grain size.

These nine values are averaged and one value is reported.

Appendix C

SUBSTRATE DENSITY MEASUREMENT METHOD

Appendix C

SUBSTRATE DENSITY MEASUREMENT METHOD

1. Select a representative sample of five substrates from the substrate lot.
2. Scribe and break these five substrates into quarters.
3. Select five quarters, one from each substrate.
4. Weigh each substrate quarter in air on a balance capable of resolving 0.1 mg.
5. Weighing the substrate in water is performed by suspending the substrate from the balance using a wire with an alligator clip. Weigh the wire and alligator clip on the same balance while suspending it into the beaker of room temperature deionized (DI) water.
6. Dip the substrate quarter into a wetting agent solution.
7. Immediately, without drying, fasten the alligator clip onto the substrate quarter. Suspend the wire and substrate from the balance as in step 5 such that the substrate is not touching the beaker during weighing and that the clip and wire are immersed the same distance into the DI water as before. Check for the absence of bubbles. Weigh the substrate quarter.
8. Subtract the weight of the wire and alligator clip (step 5) from the combined weight (step 7).
9. Calculate density in accordance with the following formula.

$$\text{Density} = \frac{\text{weight in air}}{\text{weight in air} - \text{weight in water}} \times \text{density of water}$$

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CHARACTERIZATION OF CERAMIC SUBSTRATES,
A. Laudel, Final, February 1979.

Characterization techniques were developed to improve receiving inspection and in-process testing of 99.5 percent alumina substrates used for manufacturing hybrid microcircuits. Many of these tests have been implemented into production; other tests have been recommended for further development. The tests developed were scanning electron microscope (SEM) measurement of grain size; low angle light inspection for surface defects; non-contact thickness and camber test; dye penetrant

ELECTRICAL: Ceramic Substrate Characterization
CHARACTERIZATION OF CERAMIC SUBSTRATES;
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CHARACTERIZATION OF CERAMIC SUBSTRATES,
A. Laudel, Final, BDX-613-2057 (Rev.), February 1979.

Characterization techniques were developed to improve receiving inspection and in-process testing of 99.5 percent alumina substrates used for manufacturing hybrid microcircuits. Many of these tests have been implemented into production; other tests have been recommended for further development. The tests developed were scanning electron microscope (SEM) measurement of grain size; low angle light inspection for surface defects; non-contact thickness and camber test; dye penetrant

check for porosity and cracks; and SEM inspection of surface finish in holes. The effect of raindrop (variation of light transmission through substrates) and polished high spot defects were determined.

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