

# A Comparative Study on the Production and Characterization of Thin Alumina Wafers for Electronic Circuit Substrate Applications

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## Abstract

Alumina (Al<sub>2</sub>O<sub>3</sub>) based thin (<500µm) wafers used as electronic circuit substrates are currently imported to Turkey. In this study, an examination of the structural and electrical features of the commercial substrates was conducted. Afterward, local production of these wafers with comparable properties was investigated for rapid and mass production. In the study, 750 µm thick green bodies were produced by dry pressing method and displayed self-supporting properties. The samples were sintered at 1550°C for 90 minutes, with a support mechanism to prevent bending of green bodies during sintering. Sintered alumina substrates were surface treated using diamond solution and Lapping workbench, and surface parallelism and smoothness were achieved with 1% precision. Abrasive diamond sizes and usage of lubrication amounts were optimized for desired surface smoothness, and then various methods were tried for surface cleaning to achieve desired surface quality. Both reference and experimentally produced wafer samples undergone a series of analyses; phase analysis by X-ray diffraction, microstructural examination by scanning electron microscope, surface roughness determination by scanning probe microscope, and dielectric properties by frequency-dependent electrical measurements. As a result of this study, substrates with a thickness of 250-500 µm met all the desired structural qualities and their properties were determined.

## Elektronik Devre Altlığı Uygulamaları için İnce Alümina Plakaların Üretimi ve Karakterizasyonu Üzerine Karşılaştırmalı Bir Çalışma

## Özet

Elektronik devre altlığı olarak kullanılan ince (<500µm) Alümina (Al<sub>2</sub>O<sub>3</sub>) plakalar halihazırda Türkiye'ye ithalat yoluyla tedarik edilmektedir. Bu çalışmada, öncelikle ticari altlıklarının yapısal ve elektriksel özelliklerinin incelenmesi yapılmıştır. Daha sonra bu altlıkların kıyaslanabilir özelliklerde, hızlı ve seri bir şekilde yerel üretiminin koşulları araştırılmıştır. Çalışmada kuru presleme yöntemi ile 750 µm kalınlığında yaş bünyeler üretilmiş ve bunlar kendini taşıyabilme özelliği sergilemiştir. Numuneler, sinterleme işlemleri sırasında yaş bünyelerin bükülmesini önleyecek bir destek mekanizması ile 1550°C'de 90 dakika sinterlenmiştir. Sinterlenmiş alümina altlıklar, elmas solüsyonu ve Lepleme tezgahı kullanılarak endüstriyel işleme koşullarında yüzey işlemine tabi tutulmuş ve %1 hassasiyetle yüzey paralelliği ve pürüzsüzlüğü sağlanmıştır. İstenilen yüzey düzgünlüğü için aşındırıcı elmas boyutları ve yağlama miktarları optimize edilmiş, ardından istenilen yüzey kalitesine ulaşmak için yüzey temizliği için çeşitli yöntemler denenmiştir. Hem referans hem de deneysel olarak üretilmiş altlık numuneleri bir dizi analizden geçmiştir; X-ışını kırınımı ile faz analizi, taramalı elektron mikroskobu ile mikroyapısal inceleme, taramalı prob mikroskobu ile yüzey pürüzlülüğü ve frekansa bağlı elektriksel ölçümlerle dielektrik özellikler karakterize edilmiştir. Bu çalışma sonucunda tüm yapısal isterleri ve kriterleri karşılayan 250-500 µm kalınlığında altlıklar üretilmiş ve özellikleri belirlenmiştir.

## 1. INTRODUCTION

Alumina ( $\text{Al}_2\text{O}_3$ ) ceramics are used in many branches of industry such as high temperature, crucibles, implants in bioceramics, as combustion chamber coating, and as sensors in automobile industry, as well as power modules, as a substrate for thick film, thin film, resistor arrays, ceramic circuit boards and in radio frequency modules.

Due to its outstanding dielectric properties, alumina is a technologically dominant material. Alumina's dielectric constant (K) value makes it to be considered as an alternative material to the  $\text{SiO}_2$  in microelectronic devices.<sup>1</sup> The characteristic properties of ceramic substrates, such as high corrosion resistance, light weight when compared with other ceramic substrates, low thermal expansion, stability of the substrate at high temperatures, and also the electrical properties of the alumina ceramics ranging from highly conductive materials to the semiconductors give them a main driving force to lead the substrates market.

On product type as an example, alumina substrates lead the ceramic substrates market due to its low cost as compared to other ceramic alternatives but also as the suitability of alumina substrate in many different application areas.<sup>2</sup> In thick film technology dimensional stability, thermal compatibility with components, high electrical resistivity, low dielectric loss tangent, good machinability and low-price range become important substrate properties. Whereas their usage as an insulation material requires low dielectric constant, preferred dielectric properties such as high dielectric constant, low voltage coefficient and also low temperature coefficient.<sup>3</sup>

Alumina substrates are mainly fabricated by dry pressing from binder added and spray dried powders, or by tape-casting from alumina slurries. Dry pressing is a low-cost, high production volume method to produce single wafers for substrate applications, whereas tape-casting should be used when multilayer structures are required.<sup>4</sup>

The main aim of this study is the determination of a production procedure for high purity Alumina based thin ( $<500\mu\text{m}$ ) wafers which are used as electronic circuit substrate. These substrates are currently imported to Turkey. In this study, it was our primary intention to examine the structural and electrical features of these imported industrial substrates and to determine their properties as a reference for comparison with the locally produced ones. The effectiveness and optimization of dry pressing method was studied to establish the parameters for the production process that is suitable for rapid and mass production of the substrates. The sintering process and the final surface finishing procedures were also investigated in detail. Finally, structural and electrical features of the locally fabricated substrates were compared with the commercial counterparts.

In this study, structural and electrical features of commercial Alumina bench-mark substrates was investigated. Local production of similar alumina wafers with comparable properties was investigated for rapid and mass production. Properties of both substrates were compared.

## 2. MATERIALS & METHODS

### 2.1. Processing of the Wafers

The commercial benchmark substrate was obtained from Coors Tek Company (USA). Alumina powders containing binders (NM 9620, Nabaltec AG, Germany) was used for the local production of Alumina substrates by dry pressing method. The chemical composition of the powder is given in Table 1. The raw materials for alumina wafers suitable for electronic applications should have a low alkaline content to evade problems such as non-uniformity due to the volatilization of the alkaline ions, lower mechanical strength and also high dielectric losses.<sup>4</sup> At least  $>92\%$   $\text{Al}_2\text{O}_3$  content is required, but usually  $96\%$   $\text{Al}_2\text{O}_3$  content is preferred, because this provides the necessary properties such as dielectric constant, minimum dielectric loss, higher thermal shock resistance, as well as the reasonable sintering temperature of  $1500\text{-}1550^\circ\text{C}$ . The remaining part of the composition usually contains the glassy phase for better sintering and minor additions such as MgO to inhibit grain growth. The iron content should be kept to a minimum because it may cause coloring and increase the dielectric loss.<sup>5,6</sup> As it can be seen from Table 1, the alumina powders used for the local production of the wafers met all the requirements.

**Table 1.** Composition of the Alumina powder used for dry pressing.

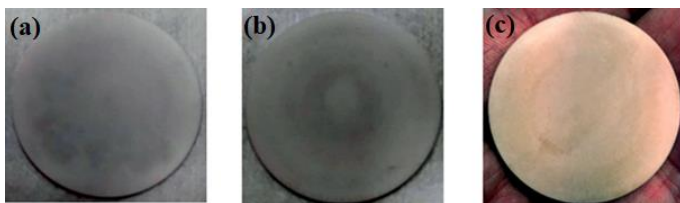
ID - %	$\text{Al}_2\text{O}_3$	$\text{SiO}_2$	$\text{CaO}$	$\text{MgO}$	$\text{Na}_2\text{O}$	$\text{Fe}_2\text{O}_3$
NM 9620	95.8	3.0	0	0.9	0.1	0.05

Steel pressing die of diameter 42 mm was used to press the powders in to disk shaped samples. The uniaxial pressure was 50 MPa. 400  $\mu\text{m}$  thick green bodies were produced by dry pressing method, and they were observed to have self-supporting properties and a good surface flatness. The green disks were sintered at  $1550^\circ\text{C}$  for 90 minutes.<sup>6</sup> The main concern during the sintering process was retaining a good surface flatness and preventing the thermoplastic deformation. A sintered thick alumina plate with a smooth surface was used as a support mechanism to prevent the bending of green bodies during sintering processes, because warpage of the wafers is known to cause problems during the metallization of the wafers during the application stages.<sup>6</sup>

The surface flatness and parallelism are of utmost importance for electronic circuit substrate applications and usually a deviation from flatness of  $> 50 \mu\text{m}/\text{cm}$  is a prerequisite.<sup>6</sup> Additionally, surface roughness of  $< 5 \mu\text{m}$  is also preferred, because larger defects may interfere with narrow conductor lines and crossovers.<sup>6</sup> On the other hand, much smoother surfaces are usually not preferred in the case of thick film substrates because that may lead to problems in the adhesion of the printed conductors and resistors.<sup>5</sup> Thus, a lapping process was integrated into the production of the substrates.<sup>4,6</sup> Lapping machines are capable of manufacturing uttermost dimensional accuracy, resulting in perfect surface finish, also setting minor imperfections of the shape right, creating truly flat surfaces normally up to  $0,1 \mu\text{m}$ .<sup>7</sup> Lapmaster Wolters Model LM15 was used in the process. A 14-micron liquid diamond slurry (Kemet Batch 237539 Code: 211011) for the lapping process and as a lubrication additive Lapmaster Wolters L30006895 /Type O oil was chosen. Full details of the lapping process were reported elsewhere.<sup>8</sup>

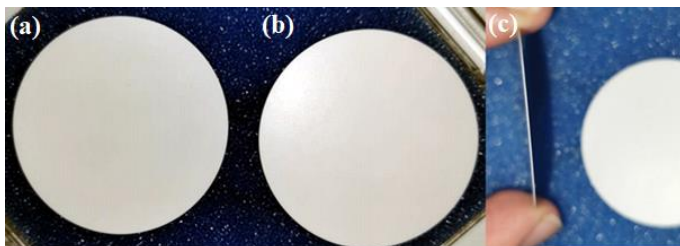
## 2.2. Cleaning of the Wafers

After the lapping process, samples were cleaned from oil - diamond slurry by physical cleaning with lubricant oil used as a solvent. After the removal of the remains of the slurry, acetone was used as a cleaning agent. At that point the substrates were fully covered with a gray colored layer. An ultrasonic cleaning was performed with water, which gives a very slight partial cleaning. Then ultrasonic cleaning was performed with acetone, but the results were not satisfactory (See Figure 1). Then the sample is placed in a furnace at 500°C to burnout any residual oils, if there were any on the surface. A pink surface formed on the sample which clearly indicated that the remaining gray particles are physically attached Fe from the surface of the steel wheel.



**Figure 1.** Surface at various stages of ultrasonic cleaning with (a) water (b) acetone, and (c) after firing at 500°C.

An acidic solution was decided to be used for the resolution of this problem as a perfect cleaner. As the steel contains other unwanted elements too, HCl acid was chosen to dissolve all the metallic impurities. A solution of HCl acid with 15 molarity was prepared, and the samples were placed in this solution. After 15 min a partial cleaning was observed. Thus, ultrasonic cleaning was decided to be used with HCl acid solution as a further modification. After 15 mins of ultrasonic cleaning with HCl acid, the samples were found to be perfectly clean with no remaining gray layer. (See Figure 2)



**Figure 2.** Surface at various stages of cleaning by (a) dipping in HCl solution, (b) & (c) ultrasonic cleaning with HCl solution.

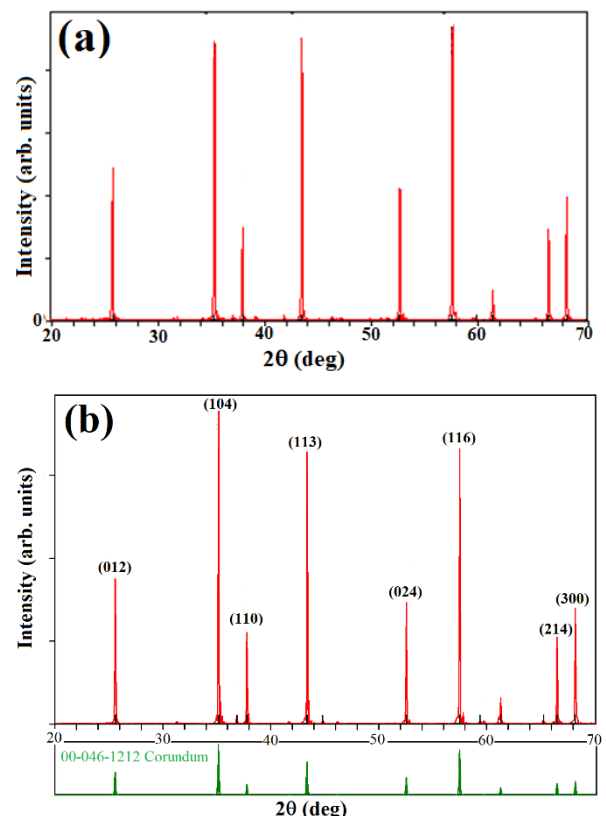
## 2.3. Characterization of the Wafers

Crystal phases of ceramic powders were determined by using an X-Ray Diffractometer (XRD). Samples were smashed into powder like form for XRD analysis. X-ray diffraction (Bruker D8 Advance, Germany) with Cu-K $\alpha$  radiation in two-theta range of 20°-90° was used to determine the crystal structure. The microstructural features and average dimensions of the grains were determined using scanning electron microscopy – SEM (XL 30 SFEG, FEI, USA). For this purpose, ceramic specimens were broken into two pieces where one half was used for investigation of the fracture surface, whereas the other half was polished and then heat treated for thermal etching. As Al $_2$ O $_3$  is known for its high corrosion resistance, the best etching method was the high temperature thermal etching, which revealed grain boundaries in a polished sample by the formation of grooves at the intersections of

Al $_2$ O $_3$  grain boundaries. Atomic Force Microscope – AFM (NanoScope IV, Veeco Digital Instruments, USA) was used to determine surface roughness of the specimens. The alumina wafers were cut into small (10 mm x 10 mm) square pieces using diamond cutting wheels for dielectric measurements. Parallel surfaces of the samples were coated with air-dry silver electrode. Relative permittivity ( $\epsilon_r$ ) and loss tangent ( $\tan\delta$ ) values of the samples were measured with an inductance-capacitance-resistance - LCR meter (3532-60, Hioki, Japan) in a frequency range of 100 Hz to 1 MHz.

## 3. RESULTS AND DISCUSSION

The X-ray diffraction patterns given in Figure 3 indicated that both the commercial benchmark and the in-house produced ceramics were pure alumina within the detection limits of the XRD equipment. All the peaks in the diffraction pattern were identified as the peaks belonging to the Corundum structure (JCPDS Card #: 00-046-1212). The main difference between the two samples were the almost an order of magnitude higher peak intensity of the in-house produced ceramic, probably due to higher crystallinity and larger grain size.



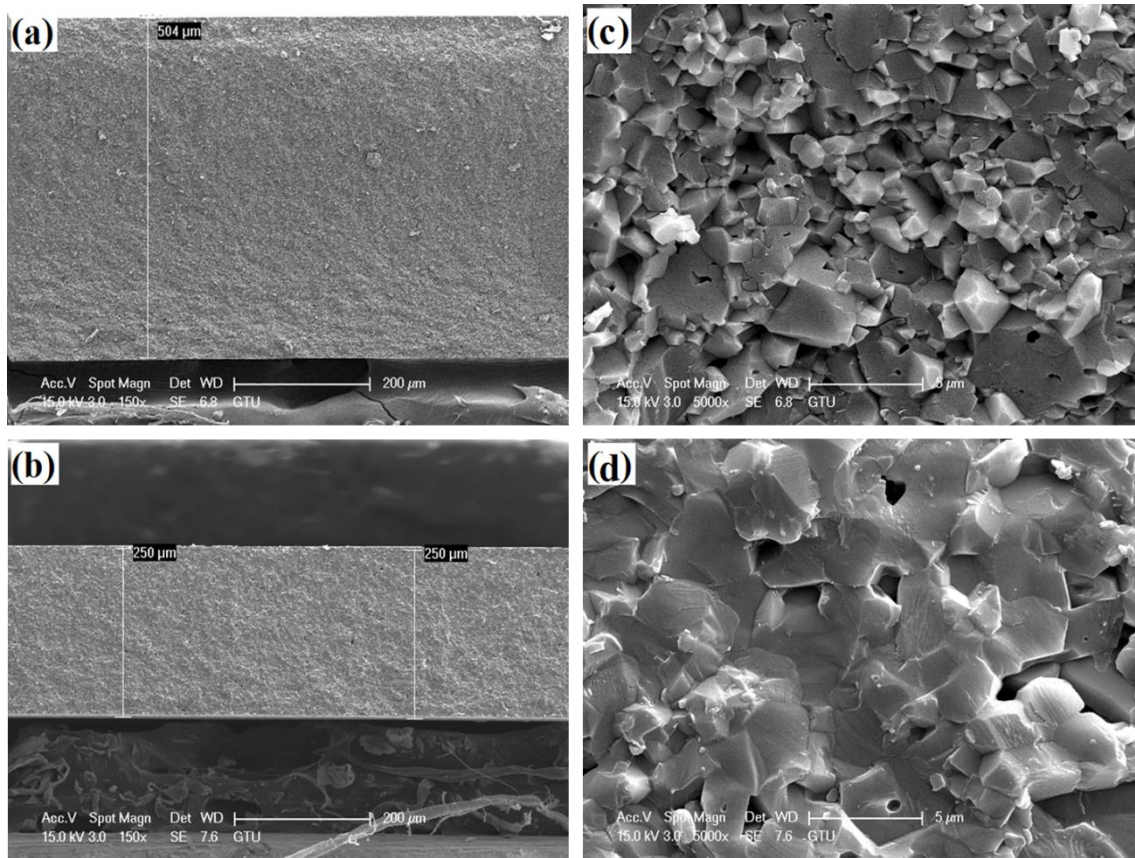
**Figure 3.** X-Ray diffraction patterns of (a) industrial benchmark wafer and (b) locally produced wafer. Peaks were indexed according to Corundum JCPDS# 00-046-1212.

The cross-sectional SEM micrographs given in Figure 4 (a) & (b) shows the entire thickness of the wafers. Although the wafers had different thicknesses (~500  $\mu$ m for benchmark and 250  $\mu$ m for the in-house produced one), their thickness was fairly uniform, and but both wafers had a fairly dense microstructure with no apparent porosity or defects.



More detailed microstructures of the fracture surfaces were given in Figure 4 (c) & (d). From Figure 4(c), the benchmark ceramic displayed a mixture of transgranular and intergranular fracture behavior, whereas

the locally produced ceramic displayed an almost fully transgranular fracture behavior, as shown in Figure 4(d), indicating a better densification and strong intergranular bonding between the grains.



**Figure 4.** Cross-sectional fracture surface scanning electron micrographs of (a) & (c) commercial benchmark and (b) & (d) locally produced wafer.

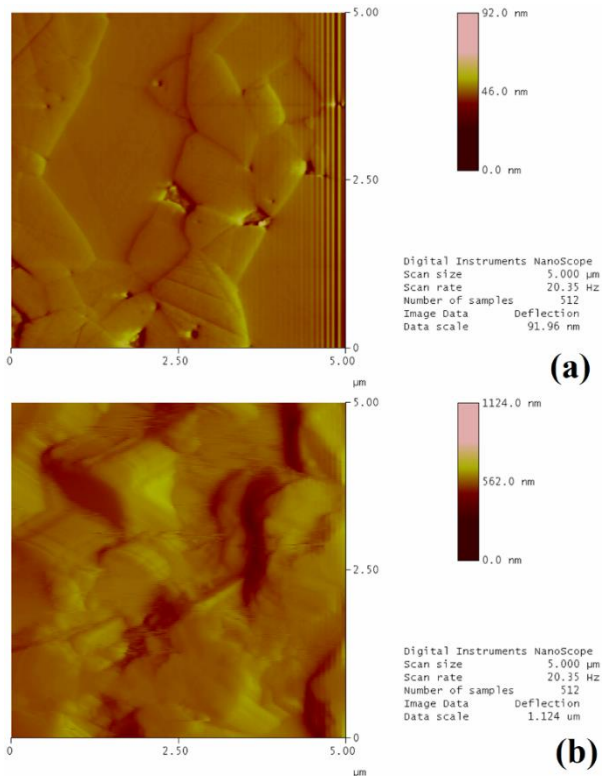
Additionally, Figure 4 (c) & (d) indicated that locally produced wafers have a larger grain size compared to the commercial counterpart. However, since fracture micrographs were not suitable for an accurate determination of the grain size, further SEM examination was conducted on polished and thermally etched surfaces (not shown here) and line intercept method was used to determine the grain sizes. According to this analysis, average grain sizes of 0.95  $\mu\text{m}$  and 1.69  $\mu\text{m}$  were determined for the benchmark and locally produced ceramics, respectively.

The surface properties, especially surface roughness of alumina wafers is of utmost importance. Especially in thick film microcircuit applications of the wafers, the adherence of the metal layers on the ceramic surface, that are deposited to provide conductive interconnections, would not be satisfactory unless the smoothness of the ceramic surface meets the requirements. If the surface is too rough, then the metallic coating becomes uneven and may peel off, whereas if the surface is too smooth like a glassy surface, then the deposited metal layer would not have sufficiently strong adherence.<sup>6</sup> Usually tape casting yields a much smoother surface compared to the dry pressed ceramics. Nevertheless, a surface roughness of less than 5  $\mu\text{m}$  was cited to be sufficient for most electronic substrate applications.<sup>6</sup> Thus, the surface properties of the commercial and locally produced alumina wafers was investigated in this study using atomic force microscopy.

Representative surface topographical images from the AFM examination were given in Figure 6. The AFM examinations were executed with 5  $\mu\text{m}$  and 10  $\mu\text{m}$  scan sizes, and scans were taken from three random positions in each sample. The average value of the surface roughness was computed from these three scans. From Figure 6(a), the commercial benchmark sample had a smooth surface with only the grains and grain boundaries creating a roughness on the surface. On the other hand, the locally produced ceramic wafer had a much rougher surface compared to the commercial counterpart, as seen in Figure 6(b).

The average  $R_{\text{max}}$  was calculated as 273.8 nm and average  $R_z$  was calculated as 158,4 nm for locally produced sample and an average  $R_{\text{max}}$  of 16.3 nm and average  $R_z$  of 9.7 nm was calculated for commercial benchmark wafer. Thus, calculated average  $R_{\text{max}}$  was 16.8 times and average  $R_z$  was 16.3 times rougher in the locally produced sample. An additional fine polishing step would help the locally produced sample to reach the benchmark sample. However, both samples are satisfying the required smoothness on the end use application, this additional polishing is only necessary to reach similar values, but it is not required for actual applications.

Finally, the dielectric constant ( $\epsilon_r$ ) and loss tangent ( $\tan\delta$ ) of both of the alumina wafers were measured at room temperature (RT) at various frequencies from 1 kHz to 1 MHz. The average values obtained from 3 different samples are presented in Table 2.



**Figure 6.** Surface topography of the (a) industrial benchmark wafer and (b) locally produced wafer, as obtained by atomic force microscopy.

**Table 2.** Average dielectric properties of alumina wafers.

ID	@ RT & 1 kHz		@ RT & 1 MHz	
	$\epsilon_r$	$\tan\delta$	$\epsilon_r$	$\tan\delta$
Benchmark	10.91	0.0013	10.95	0.0036
Local Prod.	11.31	0.0052	11.77	0.0221

These values indicate that the dielectric constant of the locally produced ceramic is comparable to the benchmark values and very close to the acceptable values (9-10), which were reported in the literature.<sup>5,6</sup> The loss values of the locally produced ceramic, on the other hand, was higher compared to the benchmark values, especially at higher frequencies, probably due to the cations that are present in the composition (Table 1). Both of the ceramics had values much higher than the ranges ( $10^{-3} - 10^{-4}$ ) reported<sup>5,6</sup> in the literature, but deemed suitable for application.

## 4. CONCLUSIONS

In this study, a production method was investigated to locally produce alumina wafers that are to be used as substrates in electronic circuits. The results of the study concluded that dry pressing of spray dried alumina powders, sintering and lapping is a sufficient and an effective production method to obtain substrates with properties that are comparable to the commercial benchmark ceramics preferred by the end user. The resultant locally produced substrates with less than 0.5  $\mu\text{m}$  surface roughness, less than 5  $\mu\text{m}$  deviation from parallelism, a dielectric constant of  $\sim 11$  and loss tangent of 0.005 (@ RT & 1 kHz) were satisfying all of the pre-determined requirements of the end user,

and also showed great potential for improvement through fine tuning of the lapping procedures.

Alumina wafers were successfully produced using a combination of dry-pressing, sintering and lapping procedures with:

- < 500  $\mu\text{m}$  thickness
- < 0.5  $\mu\text{m}$  surface roughness
- Near perfect parallelism with no warpage
- Satisfactory dielectric properties.

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## Competing interests

H.A. Ateş was an employee of ASELSAN A.Ş. when this study was conducted. Other authors do not declare any competing interests.

## Author contributions

H.A. Ateş conducted the experimental work. M. Boz contributed to the processing and lapping of the samples. N.K. Gözüaçık performed the dielectric measurements. S. Alkoy conceived and supervised the project and evaluated the results.

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