SURFACE CONTAMINATION OF ALUMINA CERAMICS BY CARBON

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Abstract:

This study presents the results of investigating the cause of contamination of aluminum oxide ceramics (Al_2O_3) in the form of brown colored stain. Contaminated ceramic surfaces were examined with the scanning electron microscope (SEM) equipped with the Energy Dispersive Spectrometer (EDS). The results show the existence of the carbon in contaminated surface that does not exist in the basic raw materials composition, not even in traces. To determine the place and source of contamination, the investigations of the production process were carried out by stages. It was found that the semi-finished product occurs in contact with carbon at two stages in the manufacturing process; by sintering semi-finished product on carbon plates and cooling with emulsion that contains carbon in traces during cutting. It was shown that contamination occurs in contact with alumina ceramics sintered on carbon plates.

1 Introduction

Aluminium oxide (alumina) ceramics, (Al_2O_3) is an exceptionally important ceramic material, which has many technological applications [1-4]. The most important properties of alumina ceramics are high hardness and strength, temperature stability, high abrasive wear resistance, and corrosion resistance at high temperatures. They are widely used due to their excellent properties [5-7]. The use of alumina ceramics in the manufacture of equipment in digital technologies is in an increase today. The production process of alumina ceramics, used as semiconductors in the manufacture of equipment in digital technologies, consists of preparation aluminium oxide powder, pressing, machining, sintering at high temperatures and finishing. Alumina ceramics with 99.8% purity is formed with cold isostatically pressed (CIP)-Al_2O_3. The reduced volume, diameter and desired shape are obtained after machine-pressing. Alumina ceramics sample is sintered by gradually increasing temperatures over time. The maximum sintering temperature is 1620°C, while the time for performing sintering is 115 hours [8,9].

After sintering, finishing treatment is conducted by cutting, flattening the surface, cleaning and polishing. Cutting is carried out on pieces of a certain thickness, depending on the final form, along with constantly cooling with emulsion that cools both tools and ceramics. Flattening the surface of the cut pieces of ceramics is conducted first on

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the one and then on the other side due to the use of cooling emulsion. Impurities (oil, corrosion products, damaged coatings, dust, soot, ash) are removed during treatment of the products in a solution of HNO_3+HF for over 10 minutes. The required surface roughness is performed by polishing with paste Strauss STP/8/W/10H Diamond Paste 2-4, Micron W-S Heavy 10G, at three stages of certain intervals. Only paste is used at the first stage, but at the third stage, paste and polishing oil Amplex Superabrasives (WS SZ21320 Diamond Lot), are used [8,9].

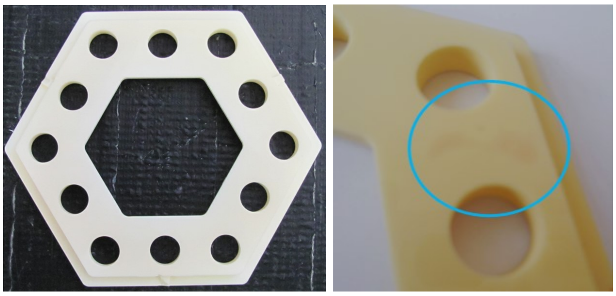
During the manufacturing process, all products exposed to strict control comply with appropriate standards depending on the type of the product. Despite this, failures on the products occur, which requires an immediate removal of the cause. Detailed examination is required considering the occurrence of certain errors that are neither known in the process nor described in the literature. The occurrence of contamination at the alumina ceramics surface can appear because of a few different factors [8,9], primarily it can be derived either from aluminum oxide powder and additives during the manufacturing process, or from deviations of the process parameters. The reason of appearance of brown coloured stains on the varying intensity surface coloration can be caused by carbon [8].

Some research has established the presence of carbon at the point of contamination that does not exist in the raw material, not even in traces, although it can be derived from the manufacturing process. The semi-finished product is in contact with carbon at two stages in the manufacturing process: during sintering of semi-finished products on carbon plates and cooling with emulsion during cutting. Investigations showed that emulsion is not the cause of contamination [8,9].

This work presents a detailed study of contamination appearance at alumina ceramics hexagons that are used for making microchips in digital technologies. Contents and distribution of carbon in a contaminated area is determined with the scanning electron microscope (SEM). The composition of a contaminated area was examined in order to determine whether the carbon is in the compounds and with which elements. With an analysis of the process, real assumptions were set about the stage of the process where the contamination occurred.

2 Materials and methods

On alumina ceramic hexagons, produced by company Applied Ceramics Ltd., Sisak, Fig. 1(a), studies were conducted.



a) b)

Figure 1. a) Panorama of alumina ceramics hexagon [10].
b) Contamination of alumina ceramics [8,10].

After polishing, contaminations were observed on almost all the products from the same batch, Fig. 1 (b), in the form of brown colored stains of varying intensity coloration on a certain number of products. Chemical composition tests were performed with inductively coupled plasma atomic emission spectroscopy (ICP-AES) [10]. The specimens from the contaminated area were taken to examine the causes of hexagons contamination with the scanning electron microscopy so that the same specimen had contaminated and uncontaminated parts. The specimens were sputter coated with palladium and gold with Emitech SC 7260 Sputter Coater, Fig. 2.

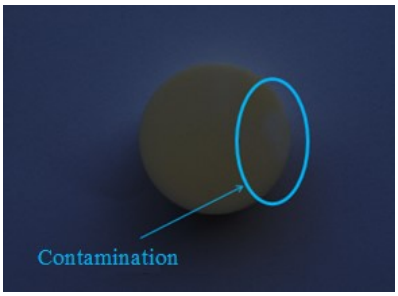


Figure 2. Sputter coated sample in a contaminated area.

Tests were conducted with scanning electron microscope Tescan Vega 5136MM. A line analysis of elements is carried out to determine the concentration profile of the particular element, and microchemical analysis in point and mapping analysis to determine the distribution of elements in selected surfaces at different magnifications.

3 Results and discussion

A visual inspection showed that the contamination occurred only on one side of the product. The test composition results of aluminium oxide powder used for making hexagons are shown in Table 1.

Table 1. Chemical composition of aluminum oxide powder, wt.%

Al_2O_3	SiO_2	Fe_2O_3	TiO_2	Na_2O	CaO	MgO
99.86	0.011	0.016	0.003	0.023	0.020	0.065

Table 1. shows that aluminium oxide powder has a composition according to the composition declared by the manufacturer [10]. The analysis has not observed any differences that could result in the appearance of stains on the ceramic hexagons.

Ćurković et al. have shown [3] that the carbon does not exist in the raw material from which the hexagons are made. Thus, contamination is formed during the manufacturing process. That is confirmed by the fact that contamination is present only on the surface. If the contamination comes from the raw materials or additives added to the raw material during the manufacturing process, contamination would be visible at the cross-section of the hexagon, on both sides of the sample, but the sample is contaminated on only one side.

Microchemical analysis (EDS method) in point, line and mapping analysis of contaminated parts of surface were performed for determination of the carbon content and his distribution in contaminated part of the sample. Fig. 3 shows the results of the line analysis of the carbon content in the tested sample. Analyses were conducted on several parallel samples.

In the sample there is the presence of carbon visible in addition to aluminum and oxygen. Other elements are presented and identified (Mg, Si, Ca, Na, Ti and Fe) in the ceramic composition, but they are not shown in figures for reasons of clarity. Fig. 3 shows the presence of the carbon in the sample 1, which is homogeneously distributed along the length of line tests. The tests were repeated on several parallel samples and almost identical results are obtained. The line analysis of elements shows no significant differences in the carbon content in contaminated and uncontaminated areas.

A mapping analysis was carried out at 500x and 1000x magnification to determine the distribution of the carbon in the samples. Results are shown in Fig. 4(a) and Fig. 4(b).

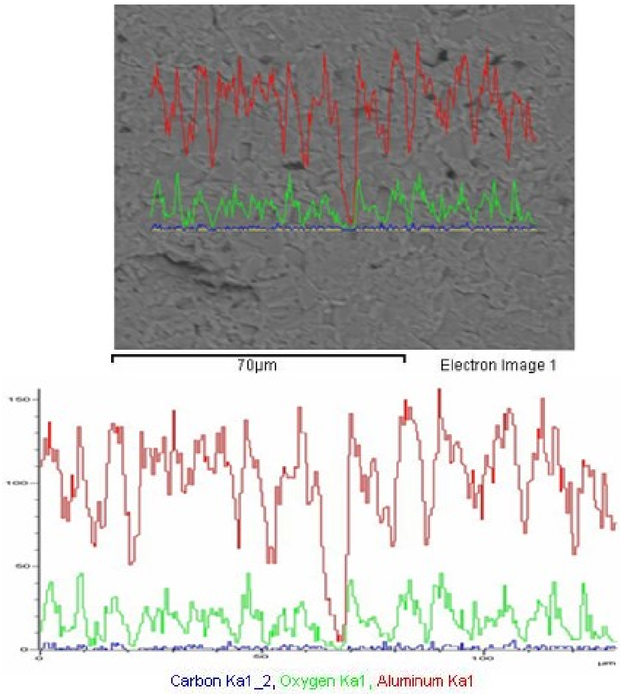


Figure 3. Line analysis of the elements in the sample 1.

At low magnification, most of the contaminated area is analyzed, Fig. 4(a). Distribution of carbon is more visible at larger magnifications, Fig. 4(b). An analysis of elements distribution has shown that the carbon existing in ceramics does not exist in compounds with other elements.

It was necessary to carry out the test method and a microchemical analysis (EDS method) in point to come to the conclusion with certainty. The obtained results are shown in Fig. 5, Fig. 6, Table 2 and Table 3. An increased content of compounds arranged in the form of points that contain carbon is visible in Fig. 5 and Fig. 6, at magnifications 1000x and 2000x. A microchemical analysis in point, shown in Table 2 and Table 3 presents that the sample surface is contaminated with carbon particles located at the surface.

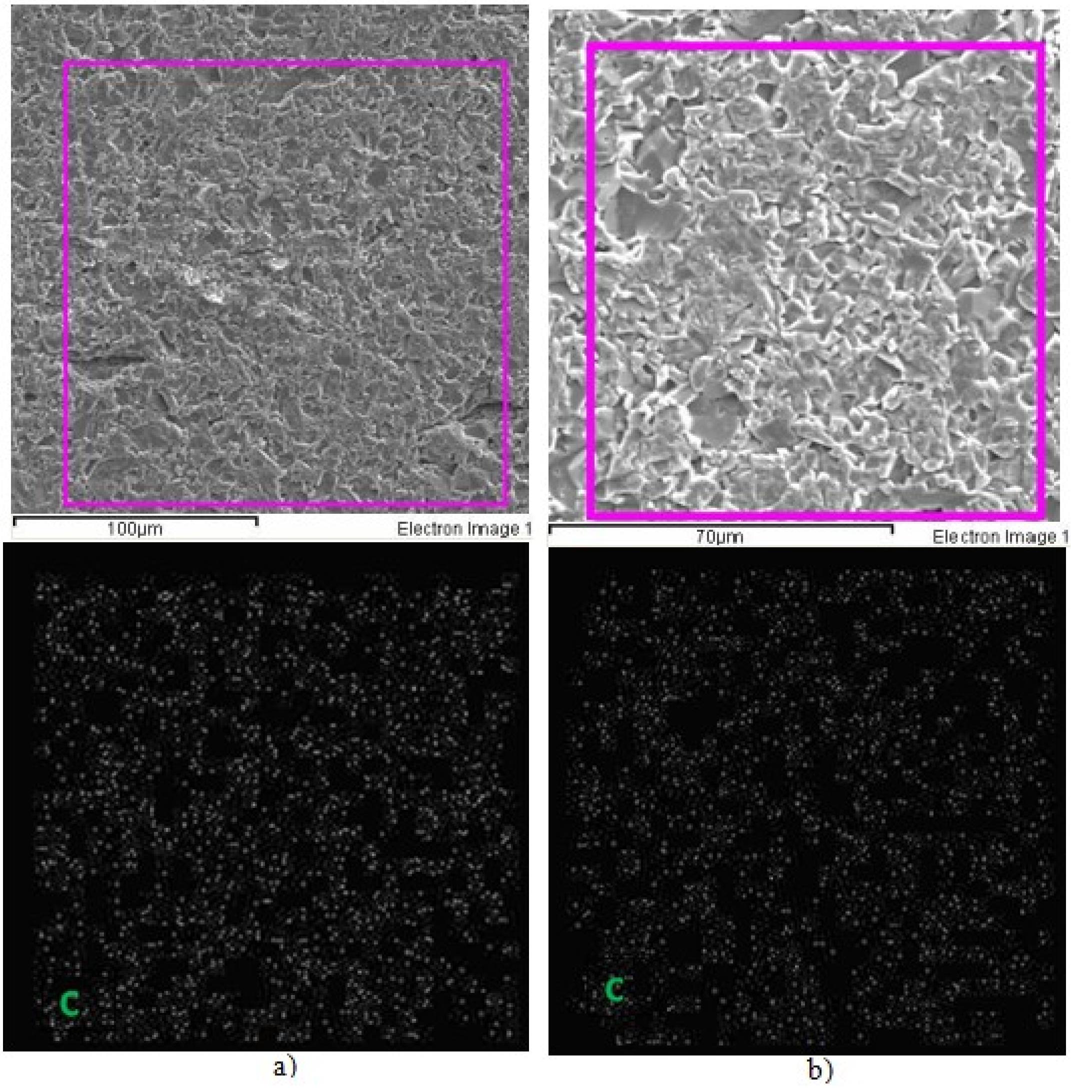


Figure 4. a) Distribution of carbon in the sample tested at magnification 500x
b) Distribution of carbon in the sample tested at magnification 1000x

From the analysis of the production process follows that a semi-finished alumina product is in contact with the carbon at the first stage of sintering ceramics. During sintering of ceramics, semi-finished products come into contact with carbon plates on which the samples are placed in the oven. Considering sintering time and sintering temperature, time is considered to be relatively long, and the temperature high, whereas carbon contamination may come from the carbon plate. The fact that the contaminated surface is formed on one side of the hexagons indicates that it occurred at this stage. Cutting of the semi-finished products into parts comes at the second stage in the production where the semi-finished products from alumina ceramics come into contact with the carbon. At this stage of the process, the hexagon is cooled with emulsion containing carbon. There is a low risk of contamination at this stage because the hexagons are heated to 1140°C where organic compounds evaporate from the ceramics. In that case, carbon disappears from the emulsion. If the contamination came from emulsions, it should be visible on both sides of the hexagon since the emulsion comes in contact with both sides of semi-finished products. But earlier tests have shown that contamination of alumina ceramics does not come from emulsions [5].

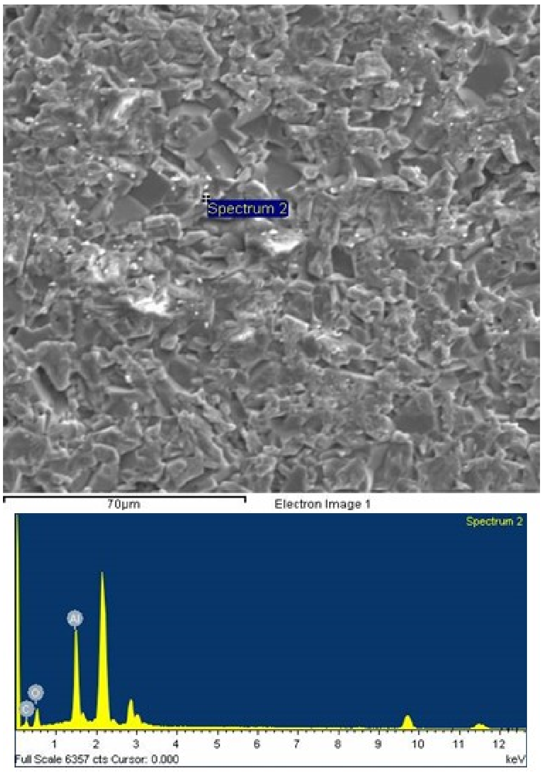


Figure 5. EDS spectrum of elements in the sample 2, magnification 1000x

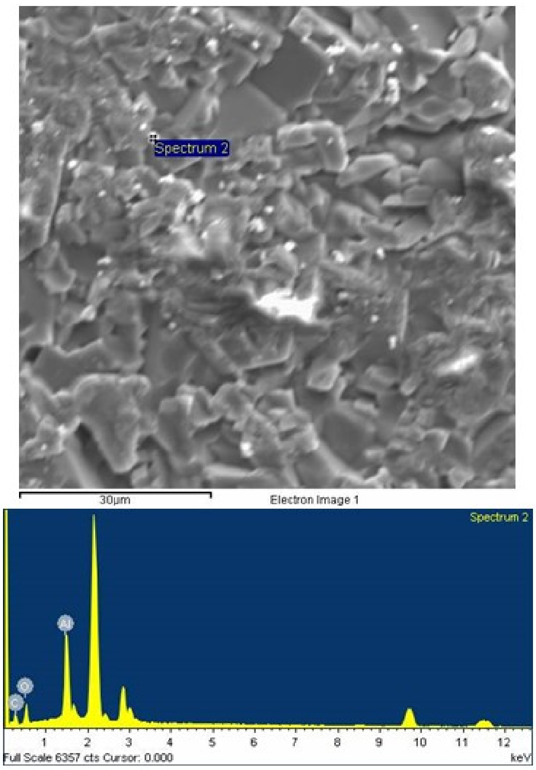


Figure 6. EDS spectrum of elements in the sample 2, magnification 2000x

Table 2. Aluminum, oxygen and carbon percentage determined by microchemical analysis (EDS method) in point on the sample 2, magnification 1000x

Element	Sample 2 1000x Normalized content [wt.%]
Al	33.92
O	37.05
C	29.03
Σ	100

Table 3. Aluminum, oxygen and carbon percentage determinated by microchemical analysis (EDS method) in point on the sample 2, magnification 2000x

Element	Sample 2 2000x Normalized content [wt.%]
Al	28.24
O	37.41
C	34.35
Σ	100

4 Conclusion

Contaminations in the form of brown stains of varying intensity coloration were observed during the manufacturing process of alumina ceramic hexagons. A detailed chemical analysis of the alumina powder has showed that there are no deviations from the prescribed chemical composition. Final hexagon products have not exhibited any deviation in chemical composition. None of the elements normally found in traces do not have an increased content.

A detailed structural analysis was conducted with the scanning electron microscopy. Carbon was to occur in the samples in contaminated parts but according to a detailed study of the chemical composition in this ceramics, carbon was not detected, not even in traces. The microchemical analysis (EDS method) in point at higher magnifications confirmed that in the samples there were particles that besides aluminum and oxygen contained mostly carbon.

A detailed analysis of the process showed that alumina ceramics occurred in contact with the carbon at two stages: by sintering ceramics at carbon plates and by cooling with emulsion during cutting the ceramics. In the phase of cutting ceramics into parts, hexagons came into contact with emulsion on both sides of the samples but contamination occurred only on one side of the hexagons. It can be thus concluded that contamination does not appear at this stage. However, in the early stage of sintering of semi-finished products from alumina ceramics, hexagons came into contact with carbon plates on only one side. Therefore, it is to be assumed that the contamination of hexagon surface has occurred from carbon plates.

Clearly, the surface contamination of alumina ceramic hexagons has come from carbon plates. So, in order to avoid contamination, particular attention is to be paid to the quality of carbon plates, especially at the early stage of sintering of alumina ceramic hexagons, but also at the beginning of their use.

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