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Shot Peening in Structural Ceramics

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Abstract

Car and aircraft manufacturers commonly use shot peening technique to modify material surface layers and improve the strength of metal components. As it occurred, the same technique can be applied for brittle ceramics. High compressive stresses up to 2.4 GPa were introduced into the near surface region of alumina and zirconia ceramics maintaining its surface integrity by ultrasonic shot peening. The dependence between diameter of tungsten carbide balls, treatment time (at constant mass of balls in the housing and vibration amplitude) and level of compressive stress introduced was determined for nano and micro sized grains of both ceramics. An increase of hardness and surface resistance to fracture with an increasing level of compressive stress was found. Surface layers of ceramics deformed by shot peening have been analysed by the classical XRD, X-ray texture test, using Euler circle and ESEM/EBSD methods.
Keywords: Shot peening, Alumina, Zirconia, Compressive stresses, ESEM/EBSD method

ŚRUTOWANIE CERAMIKI KONSTRUKCYJNEJ

Producenci samochodów i samolotów powszechnie wykorzystują technikę śrutowania w celu zmodyfikowania powierzchniowych warstw materiału i zwiększenia wytrzymałości elementów metalowych. Podobnie, ta sama technika może być zastosowana w przypadku kruchej ceramiki. Za pomocą śrutowania ultradźwiękowego wprowadzono duże naprężenia ściskające, sięgające wartości 2,4 GPa, w obszar przypowierzchniowy ceramiki korundowej i cyrkoniowej, zachowując jej powierzchniową integralność. Określono zależność pomiędzy średnicą śrutu z węglika wolframu, czasem śrutowania (przy stałej masie śrutu w obudowie i stałej amplitudzie drgań) i wielkością wywołanego naprężenia ściskającego w przypadku nano- i mikroziaren obydwu badanych tworzyw ceramicznych. Stwierdzono wzrost twardości i powierzchniowej odporności na pękanie wraz ze wzrostem wartości naprężenia ściskającego. Powierzchniowe warstwy tworzyw, zdeformowane wskutek śrutowania, zanalizowano za pomocą klasycznej techniki rentgenowskiej, metodą rentgenowskiej analizy teksturalnej wykorzystującej okrąg Eulera oraz za pomocą metody ESEM/EBSD.
Słowa kluczowe: śrutowanie, Al_2O_3, ZrO_2, naprężenia ściskające, metoda ESEM/EBSD

1. Introduction

Structural ceramics have potential for current and future materials applications which demand retained high strength at room temperature and high reliability. Both of them are dependent on the size distribution of flaws, which are formed at different stages of the technological process. Mechanisms that result in a reduced flaw population can be divided into two families: either they modify the intrinsic flaw population by altering processing characteristics and damage resistance; or they invoke mechanisms of flaw healing. A new method for ceramic surface modification was suggested by Pfeiffer and Frey [1]. Maintaining the surface integrity, high compressive stresses were introduced into the surface layer of alumina and silicon nitride by conventional shot peening using WC balls, resulting in an increase of load capacity.

In this study alumina and zirconia ceramics were treated by ultrasonic shot peening. The effect of grain size on the level of introduced compressive stresses was found. XRD, texture test and ESEM/EBSD methods were used for monitoring structural changes in the surface layer of ceramics.

2. Experimental

Alumina TMD-AR (Taimei, Japan) and zirconia TZ-3YE (Tosoh, Japan) were used for preparing ceramic samples sintered at 1350ºC and 1500ºC. As a result, two alumina ceramics having grain size: 0.97 ± 0.47 μm and 3.64 ± 1.98 μm and two zirconia ceramics: 0.095 ± 0.027 μm and 0.164 ± 0.083 μm were obtained. One surface of sintered rectangular bars was polished and given to ultrasonic shot peening using tungsten carbide balls with diameters of 1.1–0.6 mm and Stressonic® apparatus produced by SONATS (France).

The surface of the samples after shot peening was characterised by SEM and X-ray. Slow scans were prepared in the range of 24–152º 2θ, using Cu $K\alpha_1$ beam and Siemens

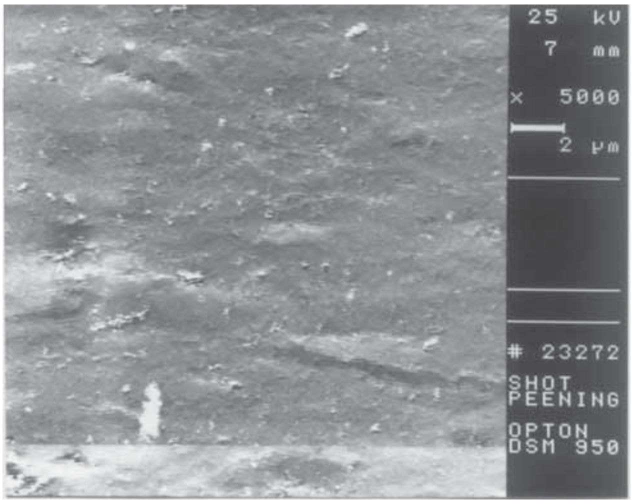
Kristalloflex 4 diffractometer equipped with Johansson quartz monochromator. Registered data were fitted by Peak-Fit program to divide diffraction lines to components and find their maximum, intensity and half breath. The $\sin^2\varphi$ method, lines (2110) for alumina and (015) for tetragonal zirconia and Fe $K\alpha$ beam were used to evaluate compressive stresses introduced into the surface layer of ceramics.

Pole figures from surface layers of ceramics deformed by shot peening have been recorded by the diffractometric method of texture measuring using Euler circle and diffractometer IRYS5 equipped with two goniometers: basic HZG4 and texture TZ5 and Co $K\alpha$ beam. Relative changes of parameters of alumina and zirconia unit cells: *a*, *c* and *c/a*, before and after shot peening, were also calculated from the data obtained in this system. The automated ESEM/EBSD (Environmental Scanning Electron Microscope /Electron Back-Scattered Diffraction) was performed to detect and map the strain fields in the surface layers of zirconia and alumina ceramics before and after shot peening. EBSD quality index (*q*) calculated on the basis of diffraction band slope was used as the local strain sensitive parameter to quantify the local distortion of the crystallographic lattices.

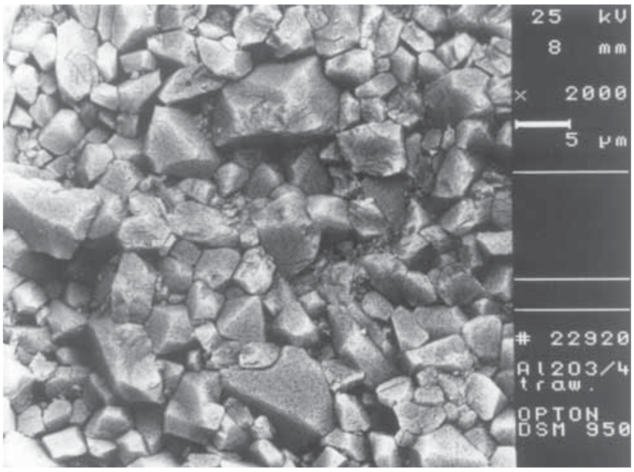
Vickers diamond indents were performed on the polished surface of samples before and after shot peening tests. Vickers hardness, H, and surface fracture toughness, K_{Ic}, were determined from the diagonal of the indent and crack length, respectively, by the equations given by Lawn *et al.* [2] and Anstis *et al.* [3].

3. Results and discussion

A simple eye observation of an initially lustrous surface of alumina and zirconia samples showed that it was getting more and more matt when the time of shot peening increased without crack formation. It seemed that hits performed by WC balls produced a localised macroscopic deformation resulting in an increasing roughness of the surface (Fig. 1). However, when a specific time of the test was crossed, different for each diameter of ball and size of ceramic grains, “islands” of the pulled out grains started to appear (Fig. 1).



a)



b)

Fig. 1. SEM image of the shot peened surface of alumina ceramics (a) and area with “island” of pulled out grains (b).

As it can be seen, compressive stress as high as 2.0 GPa was introduced into the surface layer of alumina (Table 1) and 2.4 GPa for zirconia one (Table 3) maintaining the surface integrity of ceramics. The volume of stress increases

Table 1. Length of Vickers crack in [µm] made by 98.1 N indent load on the surface of alumina ceramics as a function of shot peening time and diameter of tungsten carbide balls. In brackets (printed in bold), values of compressive stress are given.

Time of shot peening [min]	Alumina sintered at 1350ºC		Alumina sintered at 1500ºC		
	Diameter of WC balls [mm]		Diameter of WC balls [mm]		
	1.1	0.85	1.1	0.85	0.6
0	192.5 ± 6.5	192.5 ± 6.5	182.1 ± 2.8	182.1 ± 2.8	182.1 ± 2.8
1.0	173.9 ± 2.1	151.2 ± 4.3	86.5 ± 2.0 (**1.13 GPa**)	86.3 ± 4.1	
1.5	94.9 ± 3.2	140.5 ± 3.8	42.0 ± 3.4 (**1.88 GPa**)	80.3 ± 1.8 (**1.01 GPa**)	
2.0	73.2 ± 5.3 (**0.83 GPa**)	115.9 ± 4.5 (**0.33 GPa**)	Pulling out of grains (**1.93 GPa**)	74.7 ± 3.5 (**1.30 GPa**)	41.2 ± 3.5 (**1.90 GPa**)
3.0	57.6 ± 5.5 (**1.17 GPa**)	100.1 ± 4.3		54.8 ± 0.6 (**1.66 GPa**)	35.4 ± 1.8 (**2.01 GPa**)
4.0	54.0 ± 3.5 (**1.27 GPa**)	88.7 ± 3.8 (**0.45 GPa**)		Pulling out of grains	Pulling out of grains
4.5	53.6 ± 0.2				
5.0	Pulling out of grains	77.9 ± 5.3			
8.0		67.4 ± 2.8 (**0.47 GPa**)			
10.0		63.5 ± 2.5			
14.0		44.5 ± 0.5 (**1.35 GPa**)			

Table 2. Microhardness, H, and resistance to fracture, K_{Ic}, calculated from Vickers indent carried out on the shot peened (compressive stress 1.68 GPa) and not shot peened surface of alumina ceramics as a function of the load of Vickers pyramid.

Load [N]	Polished surface			Shot peened surface		
	H [GPa]	K_{Ic} [MPa·m$^{1/2}$] (Lawn equation)	K_{Ic} [MPa·m$^{1/2}$] (Anstis equation)	H [GPa]	K_{Ic} [MPa·m$^{1/2}$] (Lawn equation)	K_{Ic} [MPa·m$^{1/2}$] (Anstis equation)
98.1	18.26 ± 0.68	2.05 ± 0.08	2.53 ± 0.15	19.57 ± 0.34	8.62 ± 0.16	10.60 ± 0.19
68.6	18.26 ± 0.27	2.45 ± 0.04	3.02 ± 0.08	19.80 ± 0.42	8.18 ± 0.13	10.10 ± 0.12
49.1	18.29 ± 0.32	2.36 ± 0.05	2.67 ± 0.11	21.68 ± 0.83	7.55 ± 0.11	9.30 ± 0.16
29.4	18.42 ± 0.48	2.28 ± 0.06	2.80 ± 0.12	20.96 ± 0.42	12.70 ± 0.26	13.60 ± 0.17

with increasing time of shot peening. Nevertheless, the smaller diameter of balls the longer time of treatment has to be used to reach the same level of stress.

The time of treatment for the same diameter of balls depends also on the grain size of both ceramics. In the case of smaller grains the time of shot peening to reach the same stress is higher than for coarser ones. It shows that the applicability of energy transferred by WC ball and sensibility to stress creation in the surface layer of coarser grained ceramics (and their deformability) is higher.

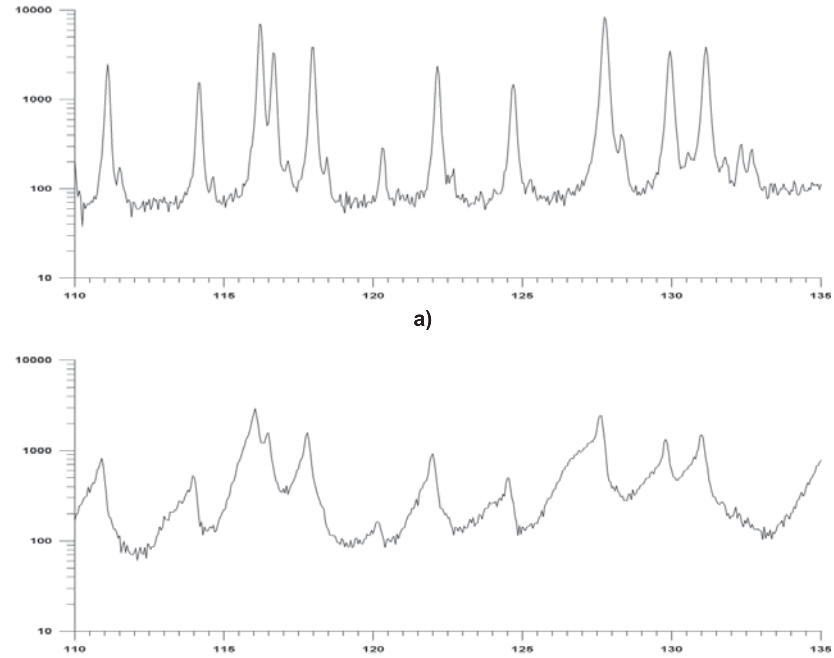
The appearance of compressive stress in the surface layer of ceramics increases its resistance to initiation and propagation of cracks introduced by Vickers pyramid. The length of Vickers cracks (see Tables 1 and 3) strongly decreases with the increasing volume of surface stresses. Contrary to the level of stress, the length of cracks is smaller for ceramics with the higher size of grains than for smaller grains at the same size of WC balls and time of shot peening. As it is shown in Table 2, compressive stress of 1.68 GPa present in the surface layer increases the fourfold surface K_{Ic} of alumina. A spectacular increase of surface fracture toughness is accompanied by 10 % increase of surface microhardness (Table 2). A similar increase of K_{Ic} is observed in the case of shot peened zirconia samples (Table 3).

Classical X-ray measurements carried out on alumina samples show a significant difference in the diagrams registered from the surface before and after shot peening (Fig. 2). X-ray peaks from the deformed surface move to lower 2θ angles and their half breadths strongly increase as compared to the same peaks obtained from the surface before the test. All peaks below 80° 2θ were fitted by a special asymmetric function. Peaks for higher angles fitted by Pearson function occurred to be the sum of two lines. One belongs to the matrix and the second to the surface layer deformed by shot peening with strongly increased half breadths, showing the decrease in crystalline size in this layer. From the X-ray data a significant increase of Al_2O_3 unit cell volume in the deformed layer is also observed. As it was found, the intensity of some peaks obtained from the deformed layer is significantly higher as compared to the matrix (Fig. 3). It could be interpreted as the effect of texturing possible only in the case of dislocation glide. No changes in grain size and grain shape distribution on the surface before and after shot peening prove that a deformation takes place only inside of the alumina grains.

X-ray results obtained for zirconia ceramics after shot peening are more complex. In the initially pure tetragonal diagram some amount of monoclinic phase appeared. X-ray

Table 3. Length of Vickers crack [μm] made by 98.1N indent load on the surface of zirconia ceramics as a function of shot peening time and diameter of tungsten carbide balls. In brackets (printed in bold), values of compressive stress are given.

Time of shot peening [min]	Zirconia sintered at 1350ºC			Zirconia sintered at 1500ºC		
	Diameter of WC balls [mm]			Diameter of WC balls [mm]		
	1.1	1.0	0.85	1.1	0.85	0.6
0	123.7 ± 13.6	123.7 ± 13.6	123.7 ± 13.6	132.2 ± 9.2	132.2 ± 9.2	132.2 ± 9.2
1.0	79.2 ± 6.6	54.6 ± 2.1 (**1.17 GPa**)	116.9 ± 9.3	86.8 ± 14.7 (**0.38 GPa**)	120.0 ± 15.1	
1.5	62.9 ± 5.6		124.8 ± 8.1	62.1 ± 6.3 (**0.60 GPa**)	118.4 ± 6.2	
2.0	54.8 ± 3.8	No cracks	76.4 ± 7.3	59.1 ± 4.3 (**0.79 GPa**)	84.0 ± 11.3 (**0.30 GPa**)	
3.0	No cracks	No cracks	85.1 ± 5.1	55.1 ± 0.9 (**0.92 GPa**)	82.9 ± 4.3 (**0.36 GPa**)	85.5 ± 8.4
4.0	No cracks	No cracks (**1.86 GPa**)		No cracks (**1.62GPa**)	81.8 ± 3.9	
5.0			79.1 ± 8.3	No cracks (**2.42 GPa**)		81.1 ± 10.3 (**0.29 GPa**)
6.0				No cracks (**2.46 GPa**)		
8.0	No cracks (**1.68 GPa**)		75.2 ± 4.2	Pulling out of grains		69.3 ± 4.7 (**0.25 GPa**)
10.0	No cracks (**1.68 GPa**)		71.6 ± 3.5 (**0.37 GPa**)			62.8 ± 2.6 (**0.53 GPa**)
11.0	Pulling out of grains					



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b)

Fig. 2. X-ray diagrams recorded from the surface of alumina ceramics sintered at 1350ºC: a) before shot peening and b) after shot peening.

peaks moved to lower angles like in alumina ceramics but broadened so strongly that overlapping of the neighbouring lines is observed (Fig. 4). In a result of the decrease of peaks amount, the strongest lines can be fitted to the pseudocubic zirconia cell, where $a_c = 2 \cdot a_t$ and $c_c = c_t$. Yet the structure of deformed layer is still tetragonal, what is proved by the presence of truly tetragonal lines: (220), (102), (212) and (104). Similarly to alumina a significant increase of tetragonal unit cell parameters was found.

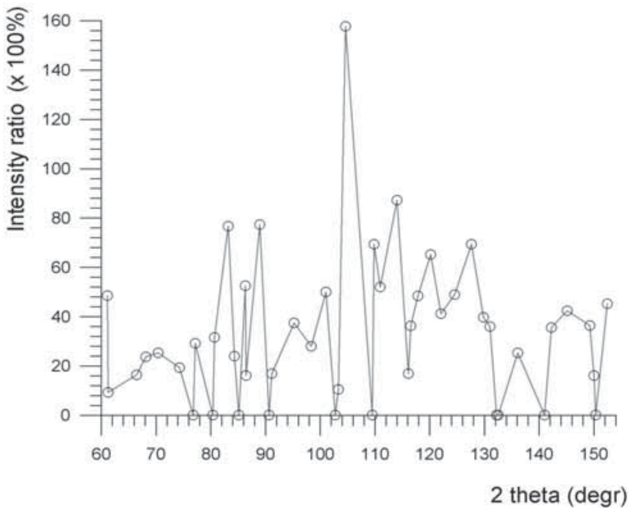

Fig. 3. Intensity ratio of the same peaks obtained from the deformed layer after shot peening with compressive stress of 1.13 GPa and the matrix of alumina ceramics sintered at 1500ºC.

As it was mentioned before, classical diffractometric measurements showed the increase of some peaks intensity obtained from the deformed layer in comparison to the same peaks of the matrix which would suggest the appearance of texture. To explain the reason of this observation surface layers of both ceramics have been tested by the diffractometric method of texture measuring using Euler circle [4]. Precise registration (1°x1°) of pole figures for polished surface and surface with maximum of compressive stress introduced by shot peening into alumina and zirconia ceramics has been done. As it can be seen from Fig. 5, differential pole figures show images typical for random distribution of grains orientation, independent of grain size of alumina ceramics. The same result was obtained for zirconia ceramics.

This proves that shot peening does not lead to the dislocation glide and texture in deformed layers of alumina and zirconia. As a result of shot peening, however, a change of parameters of alumina and zirconia unit cells and shape of statistical cell are observed (Fig. 6 and 7). As it has been found, the change of cell parameters depends on the intensity of shot peening and angular position of the sample and is responsible for the stress presence, observed in surface layers of both ceramics.

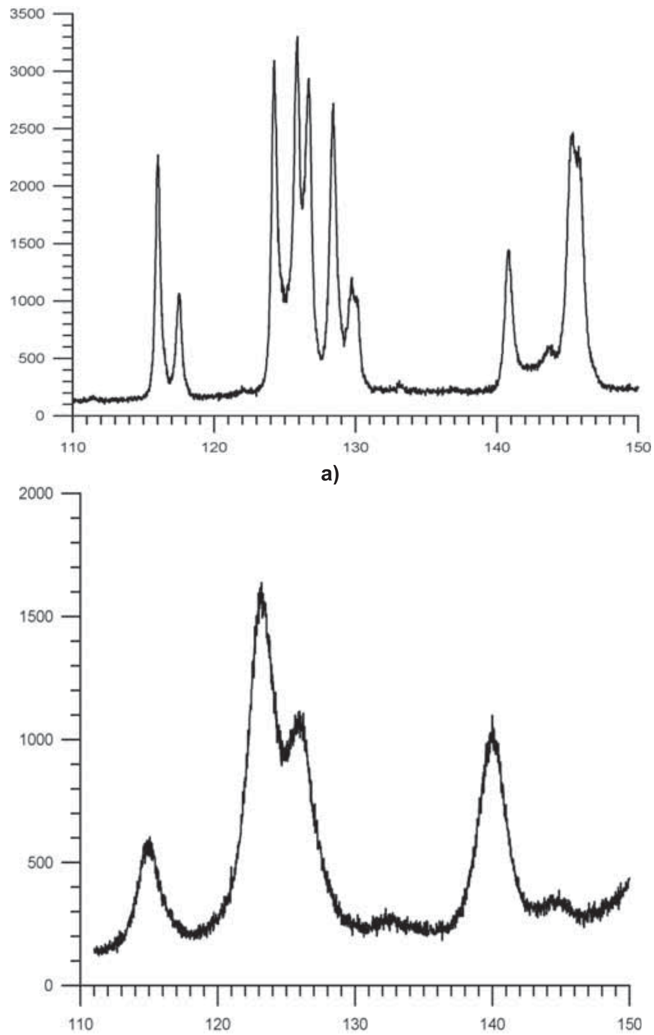

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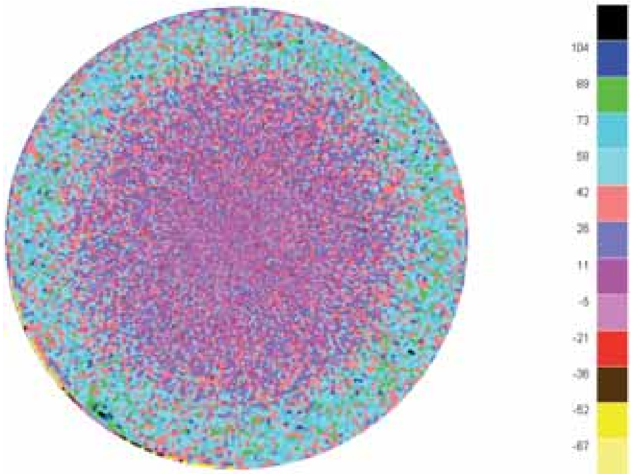
Fig. 4. X-ray diagrams recorded from the surface of zirconia ceramics sintered at 1350ºC: a) before shot peening and b) after shot peening.



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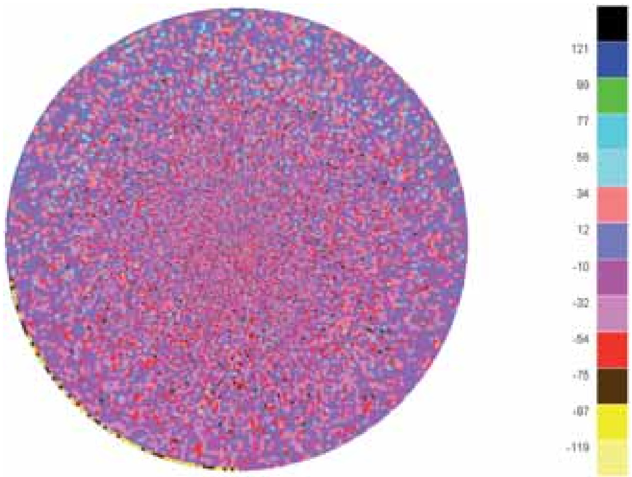
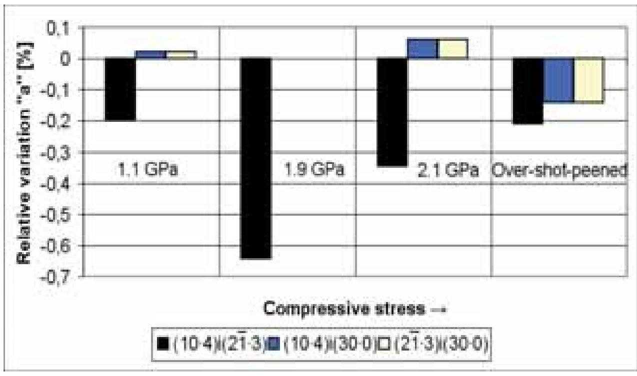


b)

Fig. 5. Differential pole figures of alumina ceramics obtained as a difference between (10-4) pole figure intensity of sample before shot peening and the same intensity of sample after shot peening with maximal compressive stress introduced: a) fine grained ceramics and b) coarse grained ceramics.

Crossing the specific time of shot peening leads to pulling out of grains from the surface layer of ceramics. It means that there is a limit of ability of ceramic materials for stress absorbing by the unit cell deformation. Fig. 6 shows that over shot peening makes *a*, *c* and *c/a* parameters come back to the values typical for a not treated sample.

To detect and map the strain fields in the surface layers of zirconia and alumina ceramics before and after shot peening the automated ESEM/EBSD was performed [4]. EBSD quality index, *q*, calculated basing on the diffraction band slope, confirmed a local distortion of the crystallographic lattices of strongly deformed surface layer produced by shot peening, increasing with compressive stresses introduced. An example of comparison of normalized *q* distributions for zirconia sample surfaces before and after shot peening is shown in Fig. 8.



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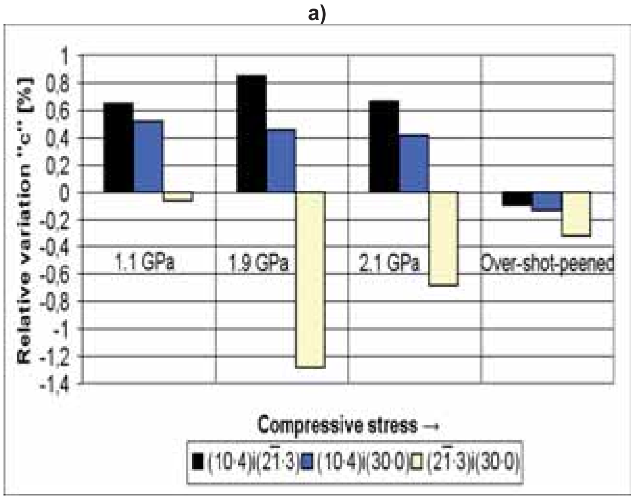

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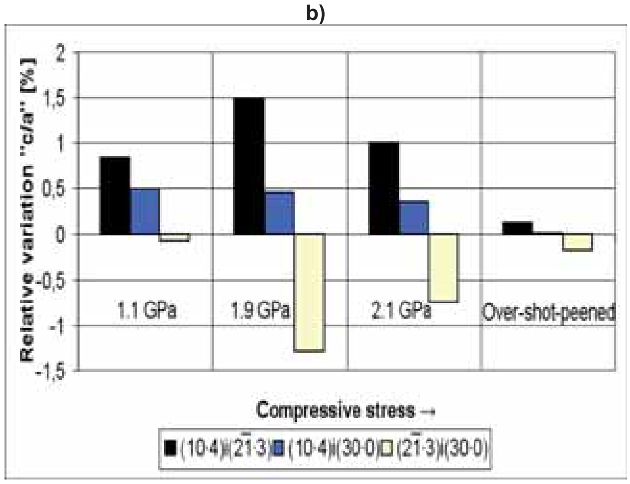

c)

Fig. 6. Relative variation of alumina cell parameters calculated on the basis of X-ray analysis for pair peaks marked in a legend: a) parameter a, b) parameter c and c) ratio c/a. Compressive stresses are marked in the graph. Right-side column graph shows data for the sample crossing the specific time of shot peening test leading to pulling out of grains.

4. Conclusions

This study showed that carefully performed ultrasonic shot peening results in introducing compressive stress into surface layer of alumina and zirconia ceramics as high as 2.0 GPa and 2.4 GPa, respectively. The presence of stress leads not only to a significant increase of ceramics microhardness but especially to a spectacular increase of surface resistance to fracture. The measurements made by the diffractometric method using Euler circle showed that with increasing time of shot peening the increased deformation of a unit cell of alumina and zirconia is observed. Crossing the specific time of shot peening leads to destruction of a surface layer of ceramics and coming back of cell parameters to the values typical for a not treated sample. The absence of texture suggested by classical X-ray measurements has

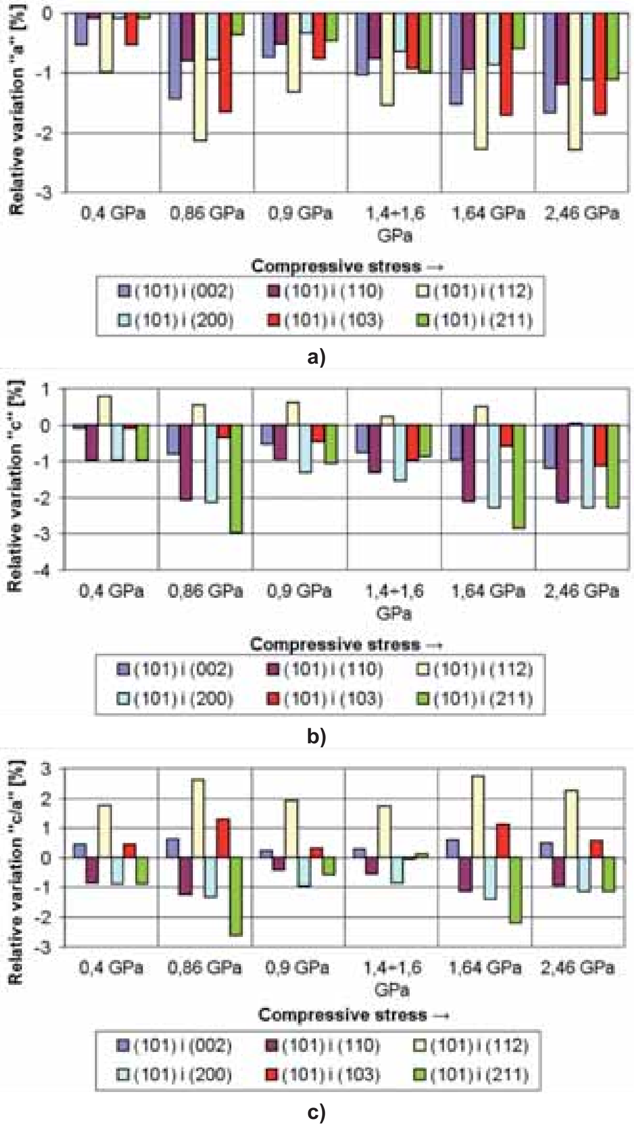

Fig. 7. Relative variation of zirconia cell parameters: a) parameter a, b) parameter c and c) ratio c/a. The change of lattice parameters was calculated on the basis of X-ray analysis for given (hkl) peaks with respect to (101) peak.

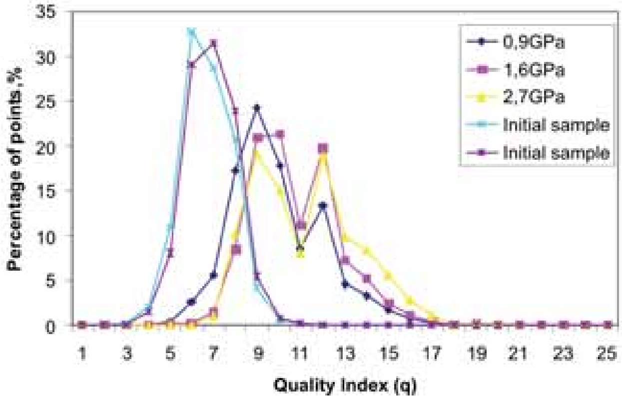

Fig. 8. Comparison of normalized q distributions for zirconia sample surfaces before and after shot peening with a different level of compressive stresses introduced.

been proved by pole figures obtained from this method. ESEM/EBSD measurements confirmed the presence of the increasing volume of defects in deformed layers of both ceramics with the increasing compressive stresses, introduced by shot peening.

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