

## Modification of alumina ceramics properties by Stressonic® shot peening

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**Abstract.** Modification of material surface layers by conventional shot peening is commonly used to improve the strength of metal components. As it occurred high compressive stresses up to 2.0 GPa may be introduced also to near surface region of alumina ceramics by this technique. Two alumina ceramics were given to ultrasonic shot peening which is a new, compact and small consuming of shot media method developed by Sonats company. This process is named Stressonic®. The dependence between diameter of tungsten balls, treatment time (at constant mass of balls in the housing and vibration amplitude) and level of compressive stresses introduced was determined. High microplastic deformation in shot peened surface layer of alumina was observed by X-ray diffraction. An increase of surface resistance to fracture of ceramics with increasing level of compressive stresses was also found.

### Introduction

Structural ceramics have potential for current and future materials applications that demand retained high strength at room temperature and high reliability. Both of them are dependent on flaw size distribution, which are formed at different stages of technological process. One type of flaw is related to the powder characteristics, forming techniques and contamination. The other type is generated during grinding. 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.

Lately, a new way of ceramics surface modification was presented by W.Pfeiffer and T.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 the load capacity. However this presentation was rather technical in character.

In our case two alumina ceramics were treated by Stressonic® shot peening. The effect of Al<sub>2</sub>O<sub>3</sub> grain size on level of introduced compressive stresses was found. XRD method was used for monitoring structural changes in surface layer of ceramics.

### Experimental

Taimei TMD-AR alumina powder (grain size of 290nm) was pressed and sintered at 1350 and 1500°C. In a result two alumina ceramics with grain size 0.97±0.47µm and 3.64±1.98µm were

obtained. One surface of sintered rectangular bars of ceramics was polished and given to Stressonic® shot peening using tungsten carbide balls with diameters of 1.1-0.6mm and Stressonic® apparatus produced by SONATS (France).

Surface of the samples after shot peening was characterised by SEM and X-ray. Using a Siemens Kristalloflex 4 diffractometer equipped with Johansson quartz monochromator and Cu  $K\alpha_1$  slow scans were prepared in the range of  $2\theta$  24-152°. Registered data were fitted by Peak-Fit program to divide diffraction lines to components and find their maximum, intensity and half breath. Parameters of alumina unit cell before and after shot peening were also calculated.  $\sin^2\phi$  method, line (2 1 10) and Fe  $K\alpha$  were used to evaluate compressive stresses introduced. Value of stress  $\sigma$  was calculated from the formula (1):

$$\sigma = \Delta\epsilon_{\phi} / \Delta \sin^2\phi (1+\nu)/E \quad (1)$$

where:  $\Delta\epsilon_{\phi}$  is a change of interplanar distance,  $\nu$  Poisson's ratio and E Young modulus.

Vickers diamond indents were done on sample-polished surface 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 equations given by Lawn *et al* [2] and Anstis *et al* [3].

## Results and discussion

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

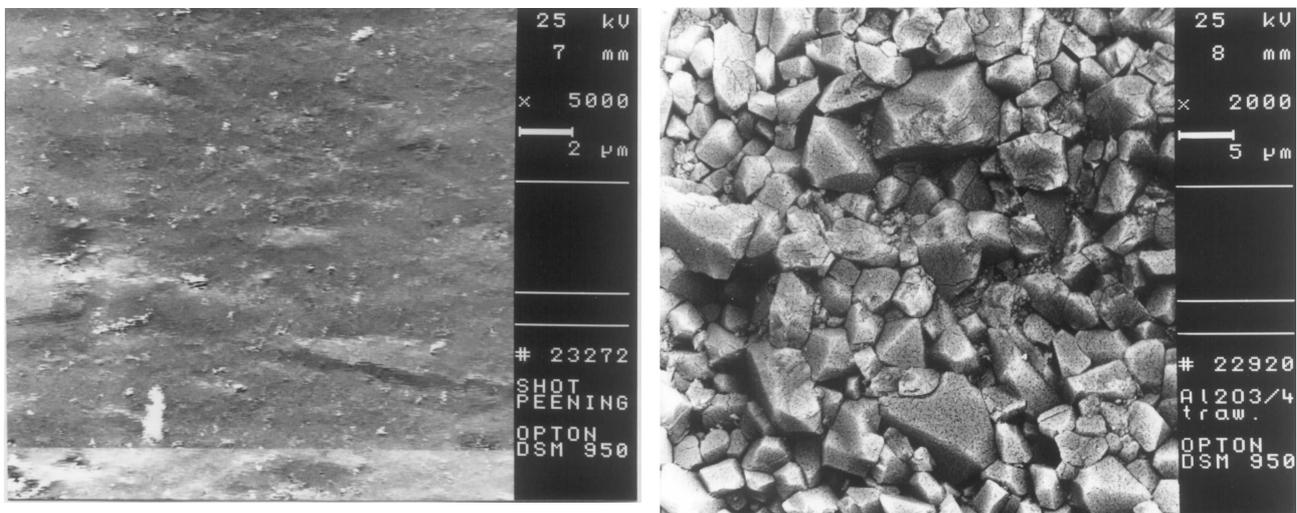


Fig.1. SEM image of the shot peened surface of alumina ceramics (left) and area with "island" of pulled out grains.

As can be seen from Table 1 compressive stresses as high as 2.0 GPa were introduced into surface layer maintaining the surface integrity of alumina ceramics. It is easy to find that stresses increased with increasing time of shot peening. However the smaller diameter of balls the longer time of treatment had to be used to reach the same level of stresses. The time of treatment for the same diameter of balls was also dependent on  $Al_2O_3$  grain size of ceramics.

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

Time of shot peening, min	Alumina sintered at 1350 <sup>0</sup> C		Alumina sintered at 1500 <sup>0</sup> C		
	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 <b>(1.13GPa)</b>	86.3±4.1	
1.5	94.9±3.2	140.5±3.8	42.0±3.4 <b>(1.88GPa)</b>	80.3±1.8 <b>(1.01GPa)</b>	
2.0	73.2±5.3 <b>(0.83GPa)</b>	115.9±4.5 <b>(0.33GPa)</b>	Pulling out of grains <b>(1.93GPa)</b>	74.7±3.5 <b>(1.30GPa)</b>	41.2±3.5 <b>(1.90GPa)</b>
3.0	57.6±5.5 <b>(1.17GPa)</b>	100.1±4.3		54.8±0.6 <b>(1.66GPa)</b>	35.4±1.8 <b>(2.01GPa)</b>
4.0	54.0±3.5 <b>(1.27GPa)</b>	88.7±3.8 <b>(0.45GPa)</b>		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 <b>(0.47GPa)</b>			
10.0		63.5±2.5			
14.0		44.5±0.5 <b>(1.35GPa)</b>			

In the case of smaller size of grains the time of shot peening to reach the same level of stresses was higher than for coarser ones. It shows that applicability of WC ball energy and this way, sensibility to stress creation of coarser alumina grains (and their deformability) is higher.

Appearance of compressive stresses in the surface layer of alumina should in turn increase the resistance of alumina to initiation and propagation of cracks introduced by Vickers pyramid. The length of Vickers cracks (see Table 1) strongly decreases with increasing volume of stresses present. Similarly to level of stress the length of crack for ceramics with higher size of grains is smaller than for smaller grains for the same size of WC balls and time of shot peening. This observation confirms earlier expectation that the higher compressive stresses in surface layer mean the higher surface resistance to fracture. Table 2 shows that increase of  $K_{Ic}$  is fourfold in the case of compressive stress presence of 1.68GPa in surface layer and should increase with increase of stress. Thickness of deformed surface layer of alumina equals 6-50 $\mu$ m dependent on shot peening time, as it was estimated from X-ray data. The higher shot peening time the bigger compressive stresses and thickness of deformed layer were observed. This observation shows that correlation between level of compressive stresses introduced by shot peening and length of Vickers cracks exists. In a result the length of Vickers crack can be used as a parameter illustrating the level of stresses present. Spectacular increase of surface fracture toughness is accompanied by 10% increase of surface alumina microhardness (Table 2).

X-ray measurements done on alumina sample with compressive stresses of 1.68 GPa show strong difference in diagrams registered from the surface of alumina before and after shot peening. The X-ray peaks made from the deformed surface not only move to lower  $2\theta$  angles but their half breadths are higher in comparison to the same peaks from alumina surface as received (Fig.2).

All peaks up to  $80^\circ 2\theta$  obtained from the surface not given to shot peening were fitted by special asymmetric function to one maximum and by standard Pearson function for higher angles. In the case of shot peened surface all peaks below  $80^\circ 2\theta$  were fitted by special asymmetric function, but they have increased half breadths. As it occurred peaks for higher angles are sum of one line fitted by Pearson function and one or two lines fitted by Gauss function with strongly increased half breadths (see Fig. 3). Gauss components are located on a lower angle side than Pearson components.

Table 2. Microhardness, H and resistance to fracture,  $K_{Ic}$  calculated from Vickers indent done on the shot peened (compressive stress 1.68GPa) and not shot peened surface of alumina ceramics sintered at  $1350^\circ\text{C}$  as a function of the load of Vickers pyramid.

Load, N	Surface not given to shot peening			Shot peened surface		
	H, GPa	$K_{Ic}$ , MPam <sup>1/2</sup> (Lawn equation)	$K_{Ic}$ , MPam <sup>1/2</sup> (Anstis equation)	H, GPa	$K_{Ic}$ , MPam <sup>1/2</sup> (Lawn equation)	$K_{Ic}$ , MPam <sup>1/2</sup> (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

In our opinion peaks fitted by Pearson function describe the intermediate layer below surface layer strongly deformed by shot peening and undeformed matrix. They not only move to the lower angles but also have bigger half breadths in comparison to the same peaks from the surface not given to the test. Peaks fitted by Gauss function describe deformed surface layer. They move to lower angles than Pearson components and have the highest half breadths, showing strong decrease in crystalline size in this layer (see Table 3). Parallel significant increase of  $\text{Al}_2\text{O}_3$  unit cell volume in deformed layer was observed as a result of increased level of point defects introduced by shot peening into alumina structure. As it was found amount of point defect in alumina structure increases with increasing time of shot peening (interplanar distance increases with test time). Intensity of some peaks obtained from the deformed layer is significantly higher. It can be interpreted as an effect of texturing possible only in the case of dislocation glide. No change in grain size distribution at the surface before and after shot peening prove that microplastic deformation takes place only inside of alumina grains.

Table 3. Parameters of  $\text{Al}_2\text{O}_3$  unit and crystalline size of alumina before and after shot peening

Type of surface	Parameters of unit cell			Crystalline size, A
	a, A	c, A	V, A <sup>3</sup>	
As received	4.75844 ±0.000038	12.99362 ±0.000130	254.7945 ±0.0066	2300
Deformed surface layer	4.76671 ±0.00366	12.99647 ±0.01122	256.8111 ±0.614	300-800

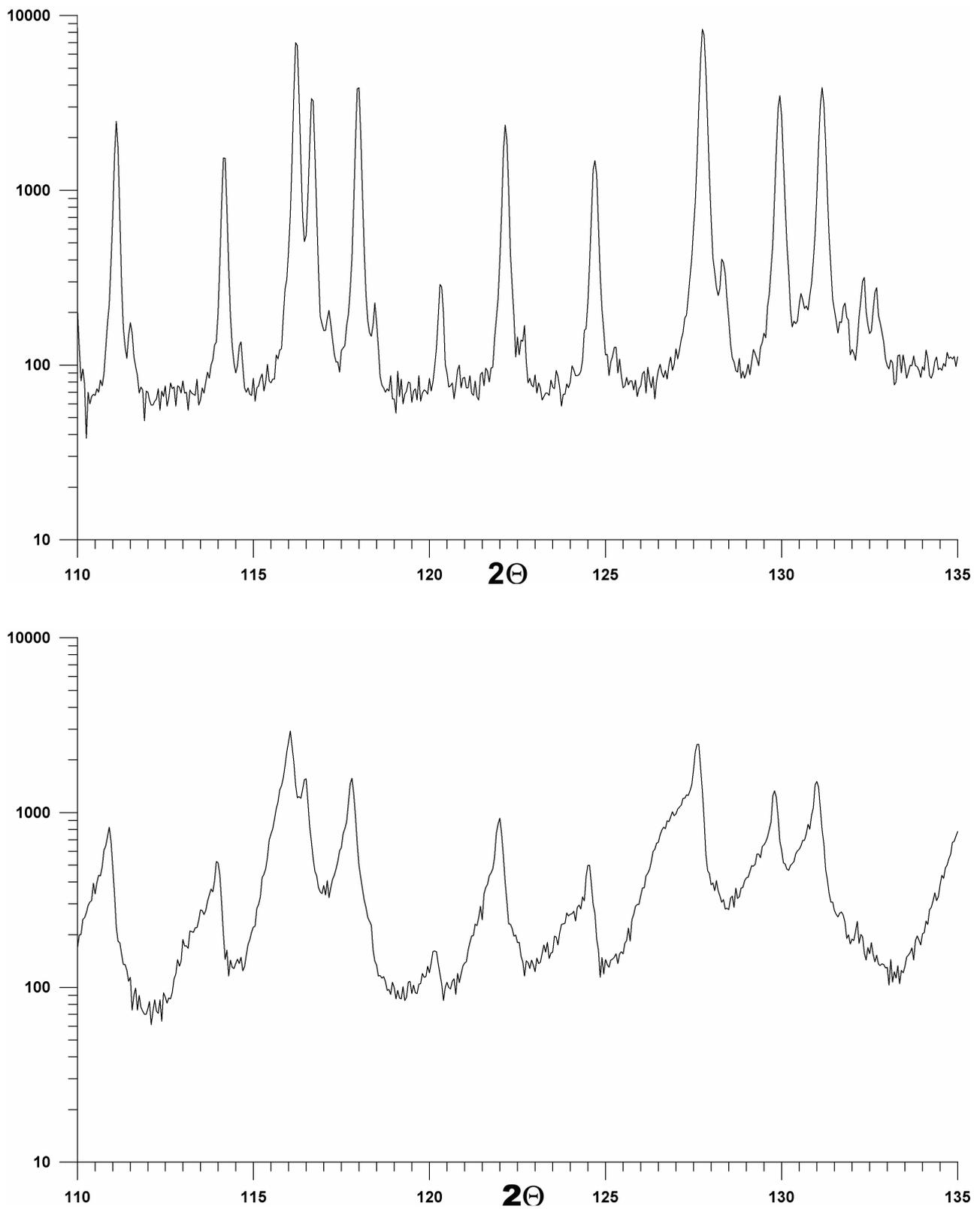


Fig.2. X-ray diagram recorded from the surface of alumina ceramics with smaller grain size before (upper scheme) and after (bottom scheme) shot peening.

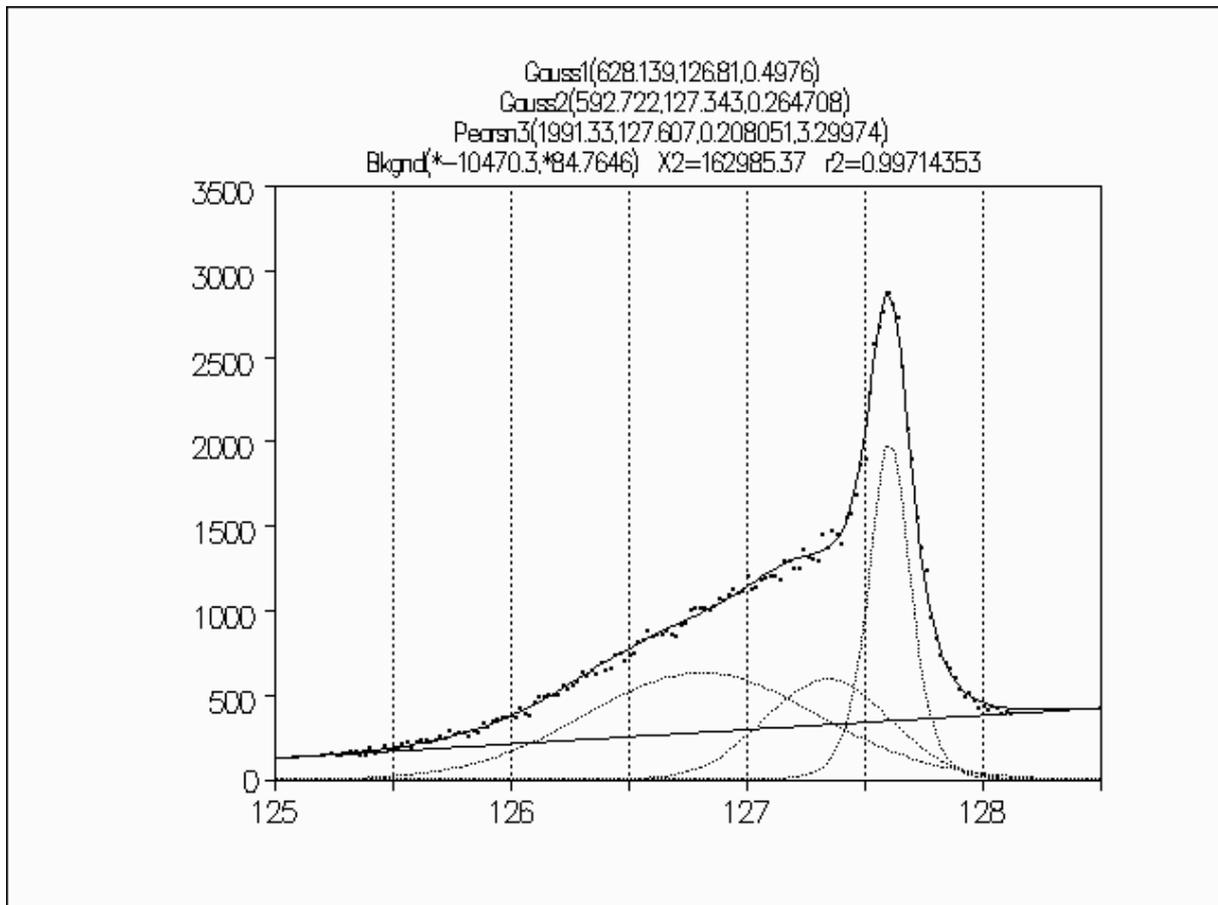


Fig.3. X-ray peak registered from alumina surface after shot peening with three maxima described by Pearson and Gauss functions.

## Conclusions

This study showed that carefully done Stressonic® shot peening results in introducing as high as 2.0GPa compressive stresses in surface layer of alumina ceramics. Presence of stresses leads not only to significant increase of ceramics microhardness but especially to spectacular increase of surface resistance to fracture. Observed deformation is microplastic in character and takes place inside of alumina grains resulting in a strong decrease in crystalline size.

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