Surface modification of plasma-sprayed alumina deposits by high-energy ion beams

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Abstract This paper reports on characteristics of alumina-type coatings deposited on steel substrates and treated with pulsed plasma streams containing high-energy ion beams (N, Ar, and Ti+N). Originally matted surfaces of the ceramic layers, which were created by plasma spraying of gray alumina, were changed into shiny ones, and splat boundaries disappeared due to the melting of a thin surface layer. The thickness of this modified layer did not exceed 1 μm. A higher content of Ti was found on the surfaces of the Ti- and N-treated samples only.

Key words alumina coatings • plasma-ion streams • plasma spraying • surface modification

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Introduction

High-intensity pulsed plasma-ion streams can be used for the modification of surfaces and subsurface layers of different materials, e.g. semiconductors, pure metals, alloys, and ceramics. The pulsed plasma-ion beams are generated by various plasma accelerators and by special facilities [1–8]. Recently, particular attention has been paid to the use of small-size laboratory devices [1, 5, 6], that are relatively simple and inexpensive.

The main aim of this work was to study the surface modification of plasma-sprayed ceramics, caused by irradiation with pulsed plasma-ion streams emitted from IONOTRON-type devices developed and operated in the IPJ [3, 7, 8]. This paper is a continuation of previous studies [5] and reports on observations of the surface morphology of the modified and untreated ceramic surfaces. The measurements have been performed by means of the scanning electron microscope (SEM) technique.

Experimental

Preparation of samples

The samples were prepared by the plasma spraying of a gray alumina on steel substrates by means of the WSP plasma torch operated in the IPP in Prague. Feedstock powder was two-phased, with a ca. 3% admixture of TiO_2. Substrate dimensions were 25×25×5 mm; and the deposited layer was ca. 400 μm in thickness. The spraying distance was 400 mm, and half of the samples were sprayed with a tube shrouding, as described in Table 1.

Table 1. Types of alumina coating and methods of surface treatment.

Type of alumina deposit	Type of high-energy ion beams from plasma discharges		
	I process	II process	III process
T (with tube shrouding)	Nitrogen	Argon	Titanium + Nitrogen
B (without tube shrouding)	Nitrogen	Argon	Titanium + Nitrogen

Treatment with high-energy ion beams

The treatment of the samples with high-energy ion beams was performed in the IPJ at Swierk. The PID (Pulsed Ion Doping) process [7] was used for the surface treatment of the samples with nitrogen- and argon-ions, and the DPE (Deposition by Pulsed Erasion) process [4] was used for treatment of the other samples with titanium- and nitrogen-ions simultaneously. During the treatment procedures a part of each sample was treated, and 25–30% of the sample surface was covered to keep it in the initial state, in order to enable possible comparative measurements.

Methods of morphological studies

The morphology of the samples was studied by means of the Scanning Electron Microscope (SEM) of the CamScan 4DV type. Observations were performed on both the untreated and irradiated, original and fractured surfaces and upon the irradiated (fractured) ones. A qualitative microanalysis was also performed upon the surfaces of all the samples.

Experimental results

Characteristics of untreated surfaces

On the untreated surfaces of the samples there were observed individual lamellar particles – "splats"-created by the flattening of molten droplets of the feedstock powder.

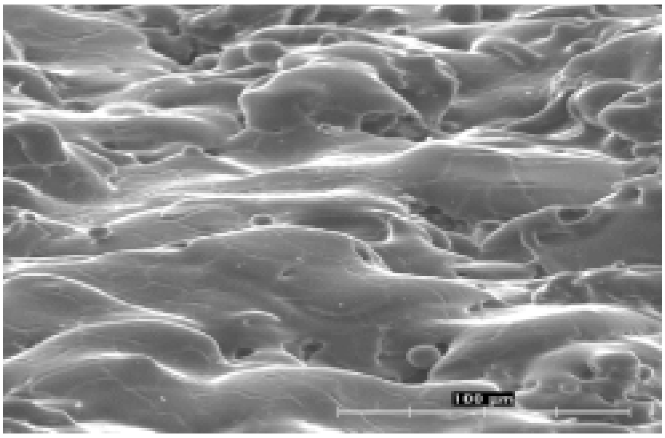

Fig. 2. Surface treated with Ti- and N-ions. Splat boundaries are no longer observed. The picture was taken at a 70° tilt.

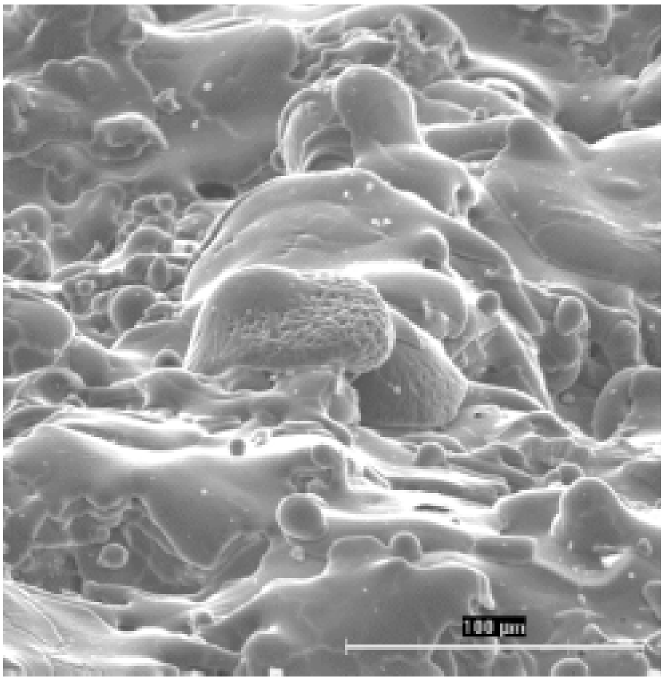

Fig. 1. Untreated surface. Splats with the dendritic structure were found only in the as sprayed material, visible in the center. A splat boundary can be seen in the upper right corner. View at a 70° tilt.

Fine dendritic structures were found in some splats, as shown in Fig. 1. Non-deformed spheres of the original powder material, re-solidified before an impact, were found occasionally. There was only a small difference between coatings sprayed with and without the shrouding tube. In the latter case a slightly larger population of the fine particles was observed. While the majority of the coating contained fine cracks, the splats formed from the TiO_2 phase remained mostly un-cracked. The cracks in alumina layers were mostly confined within each splat.

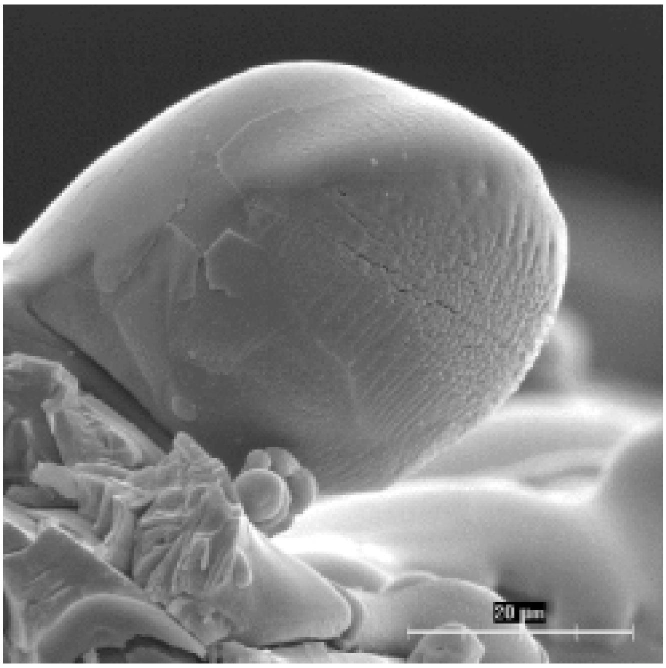

Fig. 3. Surface treated with Ti- and N-ions. The upper surface of a re-solidified sphere was smoothed by the melting, while the bottom surface retained its original relief. View at a 70° tilt.

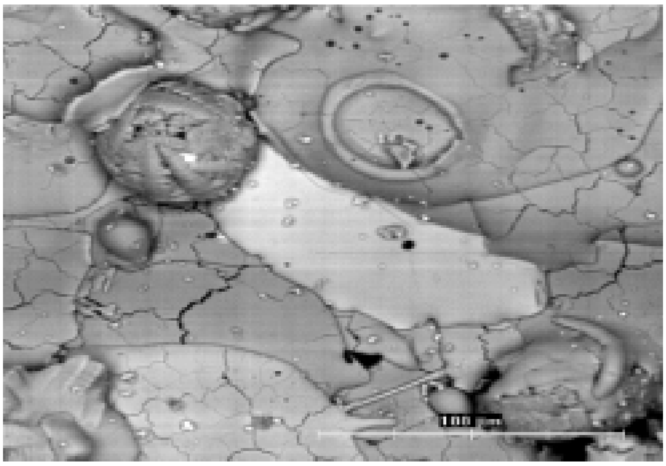

Fig. 4. Untreated surface. Each splat contains its own isolated crack network. TiO_2 particles in the center (a lighter phase) are without cracks. The picture shows a back-scattered electron image.

Upon the treated (fractured) surfaces, some columnar grains within each splat were observed. Such morphological changes can have some influence on tribological properties, but detailed investigations have been postponed to another series of laboratory measurements.

Characteristics of treated surfaces

The electron beam microanalysis demonstrated that a Ti content in the surface layer of the coating treated with Ti- and N-ions was significantly higher in comparison with that observed in the samples untreated or treated by Ar- or N-ions only. It should be noted that the original surface morphology was preserved on a macroscopic scale, but some fine features were affected, as shown in Figs. 2 and 3. In all three cases the splat boundaries were erased by the formation of a molten and newly solidified surface layer. Its thickness was smaller than the thickness of one splat. Contrary to the untreated surfaces (see Fig. 4), the network of cracks

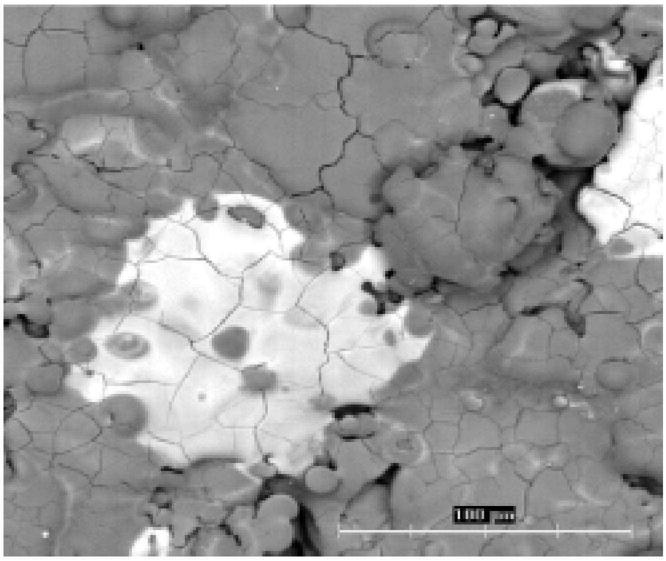

Fig. 5. Surface treated with Ti- and N-ions. Cracks are interconnected throughout the entire surface layer, also across former splat boundaries. The cracks are present also in the TiO_2 phase. The picture shows a back-scattered electron image.

extended throughout the entire surface, across the former splat boundaries (as shown in Fig. 5).

The TiO_2 phase was also cracked in this case. In the specimen treated with Ti- and N-ions the surface cracks had rounded edges as shown in Fig. 6. There are two possible explanations: initially sharp edges could have been rounded by the surface material melting or a new material could have been deposited. This has to be verified by other measurements. In other cases, the surfaces treated with the other two processes exhibited cracks with sharp edges as the original ones.

Observations of the fractured surface showed that the columnar grain structure was preserved. A thin modified

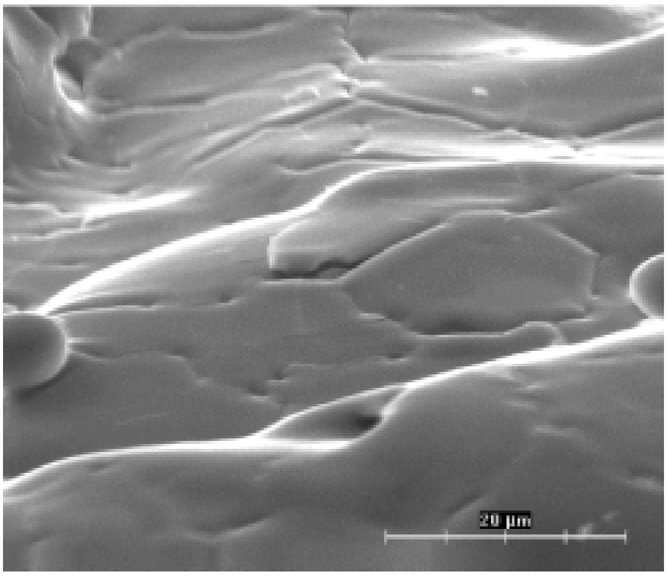

Fig. 6. Surface treated with Ti- and N-ions. The cracks with rounded edges are only seen. The picture was taken at a 70° tilt.

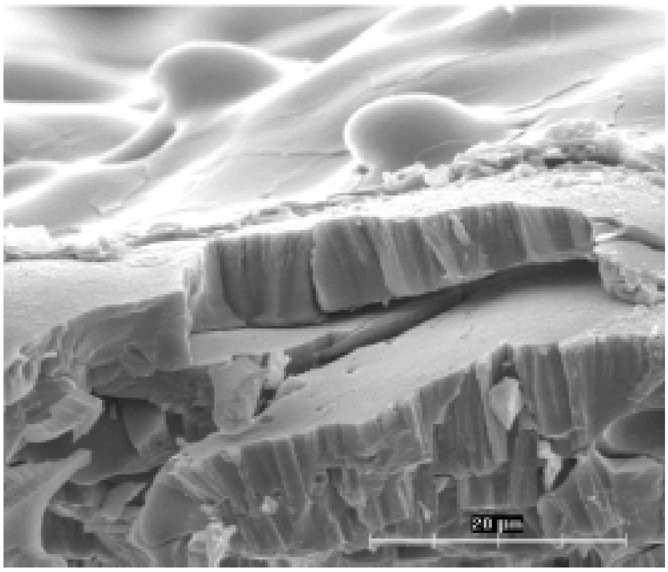

Fig. 7. Fracture surface of the specimen treated with nitrogen ions. The delamination of the top layer can be seen. View at a 70° tilt.

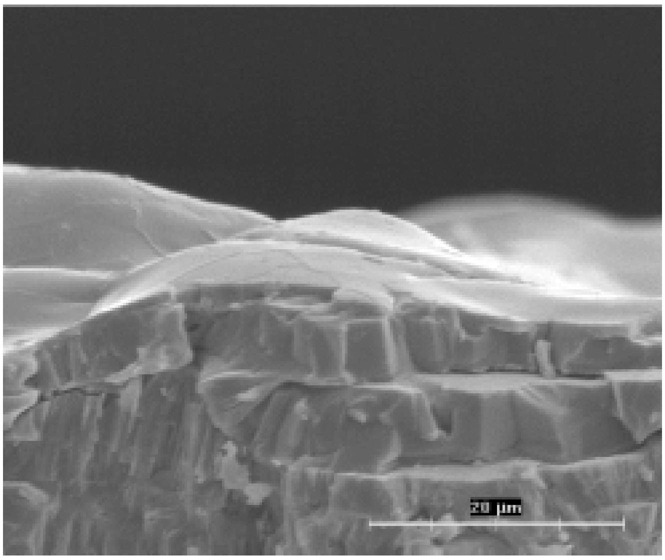

Fig. 8. Fracture surface of the specimen treated with Ti- and N-ions. A thin modified layer is observed on top of columnar grains in the surface splat. View at a 70° tilt.

layer was clearly discerned on the surface of the nitrogen-treated samples. Fracturing of the entire coating caused the delamination of this layer at the edge as shown in Fig. 7. The thickness of this layer was about 500 nm. In the argon-treated samples, such a layer could not be distinguished at all. In the Ti- and N-treated samples, a modified layer of about 800 nm in thickness was observed on the top of columnar grains in the surface splats (see Fig. 8). Unlike the nitrogen-treated surface, this layer remained adherent to the underlying material.

Conclusions

The described ion-beam treatment results in the surface modifications, which can be observed with a naked eye. An original matted surface of the ceramic layer, which was created by the plasma spraying, was changed to a shiny one and the splat boundaries disappeared due to the melting of a thin surface layer. The thickness of this modified layer did not exceed 1 µm. A higher content of Ti was found upon the surfaces of the Ti- and N-treated samples only.

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