THE MICROSTRUCTURE AND WEIBULL STATISTIC OF ALUMINA-WC PARTICULATE COMPOSITES

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Abstract

The paper presents results of investigation on the influence tungsten carbide inclusions of different size on Weibull parameter value for alumina-based particulate composites. Description of the composite reliability was done using two- and three parameter Weibull statistics and gave much better characteristic of the material. Calculated parameters of the Weibull distribution for both, two-parameter (m, σ_0*) and three-parameter distribution (m,* σ_0*,* σ_u*) were significantly different for composites with similar nominal composition. These differences could be ascribed to microstructural reasons. Microstructure analysis showed that certain differences of inclusion distribution in composite matrix strongly influence the Weibull parameters values, even if other mechanical properties (fracture toughness, mean bending strength, hardness, Young modulus) are similar.*

1 General Introduction

The manufacturing of particulate composites is the simple way to the improvement of matrix phase properties. However, usually improvement no concern every kind of property. For instance improvement of composite fracture toughness could sometimes cause decrease of their strength. Otherwise, its can lead to severe changes of Weibull statistic of the material [1].

The paper presents effect of addition of different form of WC grains into alumina matrix on strength, reliability, hardness and fracture toughness of the material.

2 Experimental

Commercially available alumina powder (Nabaltec 713-10, Germany) was used as an matrix material. Tungsten carbide additives (Baildonit, Poland) in amount of 10 vol. %, were used in form of the coarse powder (28 μm) and the finer one (5 μm). Composite powders were made by very intensive physical mixing (2 hours) of constituent phases in rotation-vibration mill in ethyl alcohol with zirconia balls of 5 mm in diameter. Such prepared powders were hot-pressed at 1650ºC for 1 hour under 25 MPa. Three type of materials were prepared for investigations – the “pure” alumina matrix (A), the composite containing coarser grains of carbide (A/WC28) and the composite containing finer grains (A/WC5).

The data for strength analysis were collected from three-point bending tests made on 30x2.5x2 mm bars.

The Weibull statistic for investigated materials was calculated applying two- and three-parameter distribution models [2].

The microstructural analyze was performed on polished composite cross-sections using scanning electron microscopy (Jeol 5400 and Nova NanoSEM, FEI Company). Tungsten carbide particle size distributions were calculated on digitalized images using free ImageJ v. 1.37 software. Calculations were conducted utilized images containing over 2000 WC grains for A/WC28 composite and over 1500 WC grains for A/WC5 composite.

The apparent density (*d*) of materials was measured by Archimedian method and compared with the theoretical on for calculation of the relative density.

The hardness (*HV*) and the fracture toughness (K_{Ic}) were measured by Vickers indentation method using Nanotech MV-700 equipment. The load was 9.81 N for hardness measurements and 29.43 N for K_{Ic} calculations.

3 Results

The intensive milling of composite powder during homogenization caused significant size reduction of WC grains below their starting size. It is clearly visible on Figs. 1 and 2. Using of rotation-vibration milling practically destroyed all WC grains present in starting powders. The final WC grain size observed in A/WC28 composite was no bigger than 4 micrometers. In A/WC5 composite maximum WC grain size was 2.8 μm.

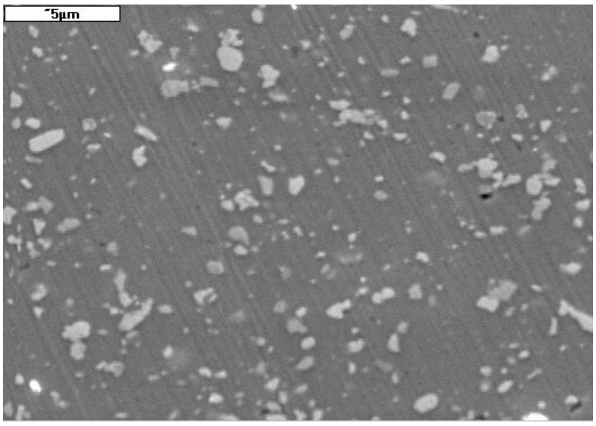

Fig. 1. A typical A/WC5 composite microstructure

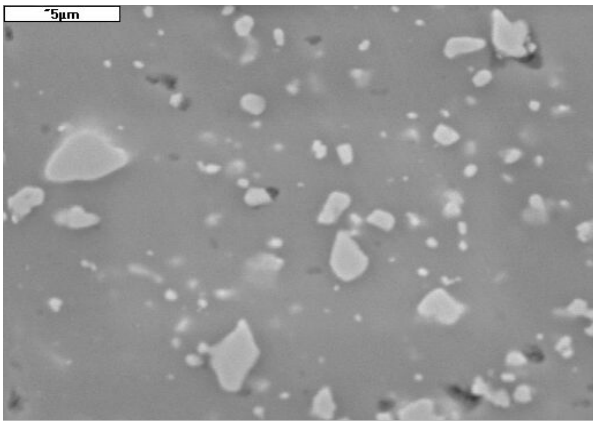

Fig. 2. A typical A/WC28 composite microstructure

Figures 3 and 4 show composites microstructures after binarization. They allowed to quantitative description of differences in particle size distribution in both composites. Conducted image analyses allowed to calculate the Feret diameters distributions.

The mean Feret diameter of WC inclusion for A/WC28 material was 0.47 μm ± 0,41 μm and 0,71 μm ± 0,69 μm for A/WC5 one.

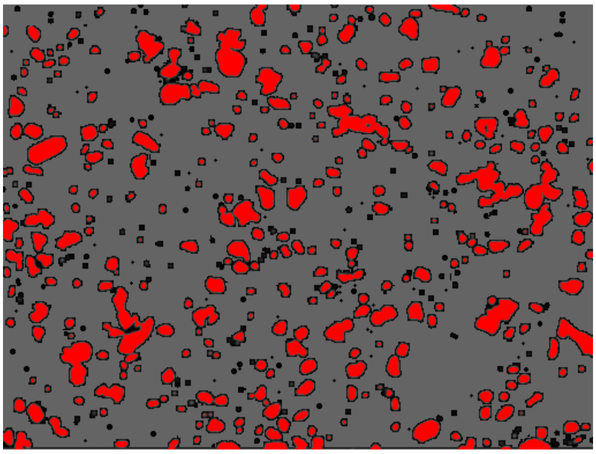

Fig. 3. A/WC5 microstructure after binarization

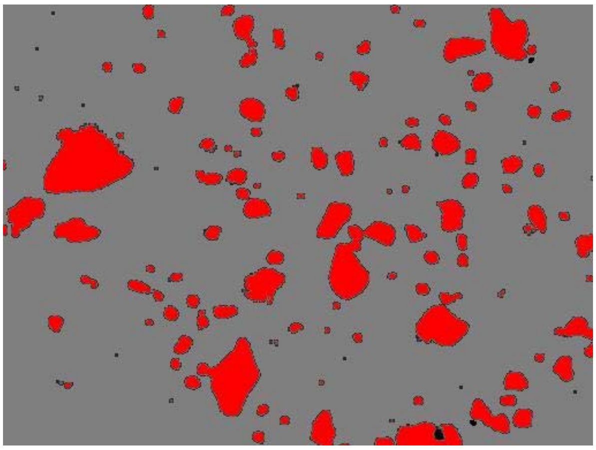

Fig. 4. A/WC28 microstructure after binarization

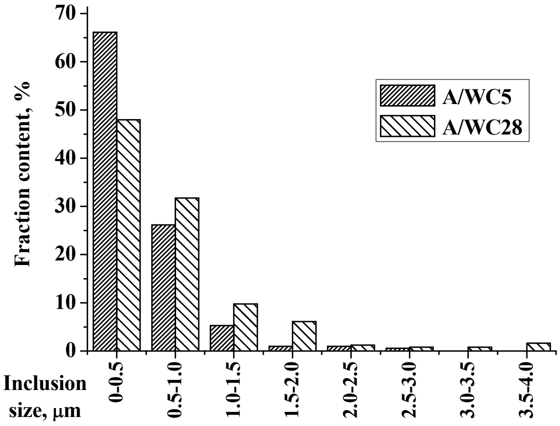

Fig. 5. Tungsten carbide inclusions distribution in sintered composite bodies - sets of inclusion dimension were divided into eight ranges

The WC inclusions size distribution in A/WC28 composite was wider than this measured for A/WC5 one. Figure 5 compares the inclusion size distribution for composites. Inclusions were divided into eight groups with 0.5 micrometer step. In A/WC5 material more than 90 % of inclusions is smaller than 1 μm. A/WC28 composites contains almost 80 % WC grains smaller than 1 μm but the fraction of 1.0 - 2.0 μm exceed 10 %. There is no grains bigger than 3.0 μm in A/WC5 material microstructure. They appear in A/WC28 composite incidentally (a few percent only).

The WC grains dispersion in the alumina matrix wasn't perfect. The detail observation of microstructures reveals many inclusion aggregates and inclusion grains in local contact (Fig. 6). For calculation there was assumed that such grains are the part of the one inclusion.

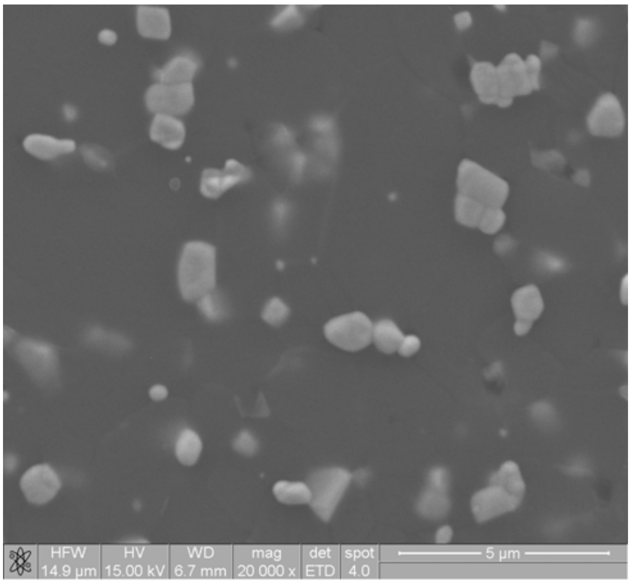

Fig. 6. A typical inclusion grains agglomeration in composite microstructure.

The difference in microstructure lead to significant difference in selected materials properties (see Table 1).

All investigated materials have similar relative densities (*d* ~99 % of theo.). Both composites showed improved hardness (*HV*) and fracture toughness (K_{Ic}). Level of the improvement was rather independent on inclusion form. Also, measured average and maximum bending strength (σ_{AV}, σ_{MAX}) values, decreased when compared with "pure" alumina material, are similar for both composites.

Table. 1. Selected properties of investigated materials
and calculated parameters of Weibull statistic

Material	*A*	*A/WC5*	*A/WC28*
d, [% theo.]	99.0	98.7	98.9
K_{Ic}, $[MPam^{0.5}]$	4.1 ± 0.1	5.63 ±0.5	5.94 ±0.5
HV, [GPa]	17,0 ±1.2	18,5 ±1.0	18.3 ±1.1
σ_{MAX}, *[MPa]*	782.09	519.19	587.81
σ_{AV}, *[MPa]*	579.20 ±117.81	417.35 ±44.16	454.60 ±87.10
m_{2p}	5.67	11.35	4.42
σ_{02p}, *[MPa]*	627.07	436.15	491.53
m_{3p}	2.17	9.31	1.90
σ_{03p}, *[MPa]*	268.48	359.55	175.81
σ_{u}, *[MPa]*	341.56	76.20	298.57

The difference between composites was pointed out when the reliability of materials were analyzed using the Weibull statistic.

Results of bending strength measurements were ranked in order from the least strong to the most strong. Cumulative distribution plots of three data sets were presented at Figs. 7-9.

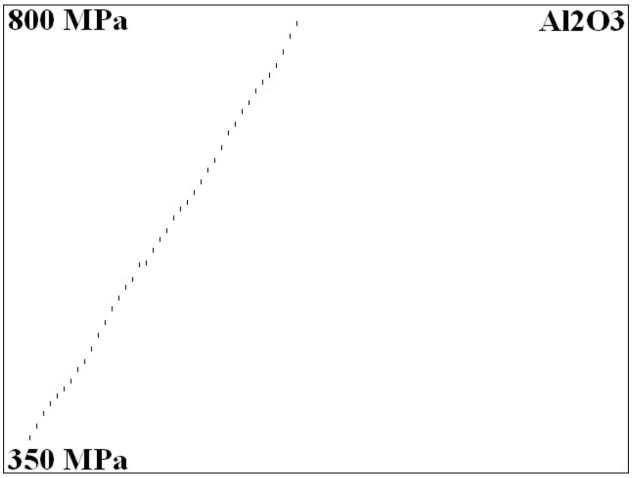

Fig. 7. Results of bending strength measurements for alumina material ranked in increasing order

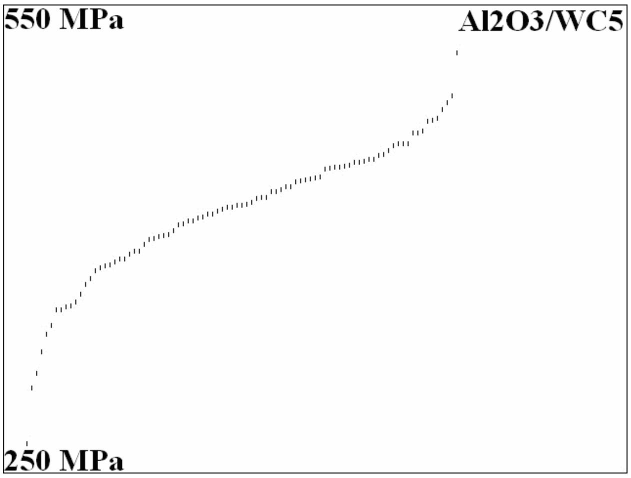

Fig. 8. Results of bending strength measurements for A/WC5 composite material ranked in increasing order

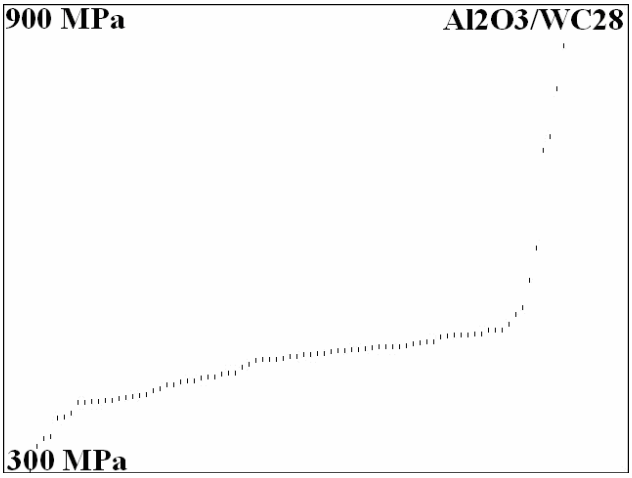

Fig. 8. Results of bending strength measurements for A/WC28 composite material ranked in increasing order

These data sets were transformed according to procedure described in [2] to plots on scales equivalent to Weibull probability model. The used procedure led to determination of parameters for two- and three-parameter models (m_{2p}, m_{3p} – Weibull parameters, relatively for two- and three parameter distribution, σ_{02p} σ_{03p} - characteristic stress values, relatively for two- and three parameter distribution, σ_u – threshold stress for three parameter distribution).

Both, two- and three parameters Weibull distributions properly described experimental data. Plots for alumina and A/WC5 composite were practically the model ones (see Figures 9 and 10).

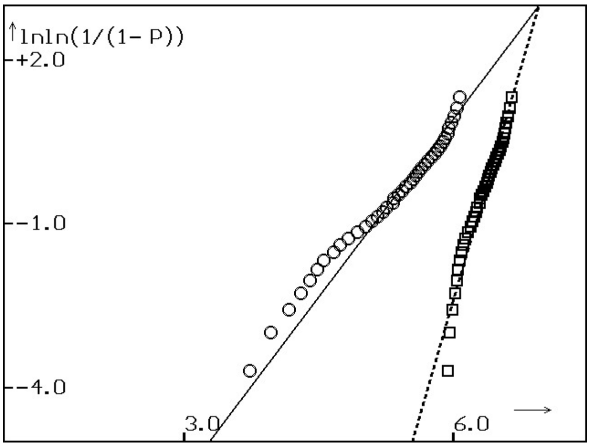

Fig. 9. The Weibull plots for alumina matrix
x axis description:
ln(σ) for two-parameters distribution (□);
ln($\sigma-\sigma_u$) for three-parameters distribution (○)

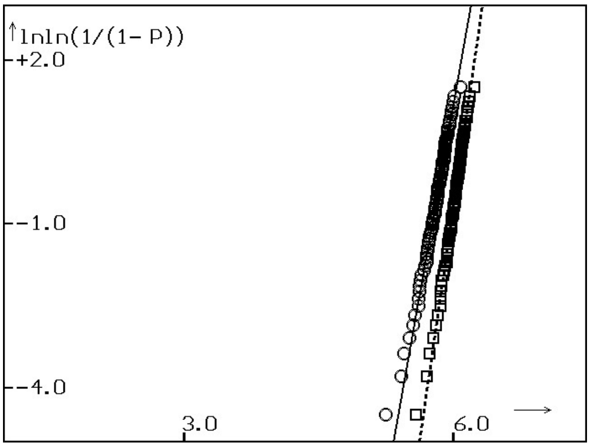

Fig. 10. The Weibull plots for A/WC5 composite (x axis description like in Fig. 3)

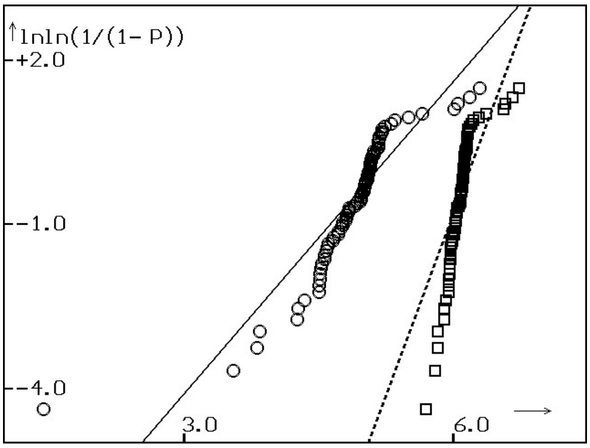

Fig. 11. The Weibull plots for A/WC28 composite (x axis description like in Fig. 9)

The different situation were observed for A/WC28 composite (Fig. 11). In this case the form of the Weibull plot suggested that WC inclusion size distribution was polymodal.

Parameters of the Weibull distribution calculated for both, two-parameter (m_{2p}, σ_{02p}) and three-parameter distribution (m_{3p}, σ_{03p}, σ_u) were significantly different for composites (see Table 1). Particularly, the difference in m parameter values for A/WC5 and A/WC28 composites is meaningful (m_{2p} = 11.35 and 4.42 relatively; m_{3p} = 9.31 and 1.90 relatively). These differences also seemed to be caused by microstructural reasons.

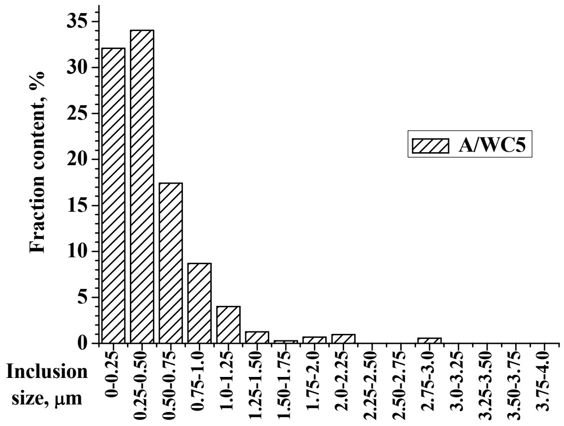

Fig. 12. Tungsten carbide inclusions distribution in sintered A/WC5 composite body - set of inclusion dimension were divided into sixteen ranges

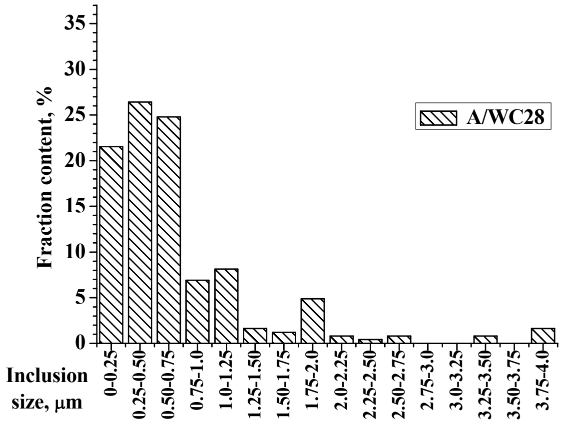

Fig. 13. Tungsten carbide inclusions distribution in sintered A/WC28 composite body - set of inclusion dimension were divided into sixteen ranges

In the next step of microstructural analyses the accuracy of inclusion distribution was increased. The data sets of inclusion dimension were divided into sixteen ranges (see Figures 12 and 13). Plots made on this base distinctly showed that the character of inclusion size distribution in A/WC5 material is completely different than this observed for A/WC28 composite.

The WC grains distribution for A/WC5 materials was, of course, narrower than this measured for A/WC28 one, but the most important was fact that inclusion distribution in A/WC28 composite was polymodal. This influenced the composite microstructure uniformity and consequently the residual stress distribution in composite material. Stresses caused by coefficients of thermal expansion (α_{Al2O3} = $7.9 \cdot 10^{-6}\mathrm{C}^{-1}$, $\alpha_{\mathrm{WC}} = 4.2 \cdot 10^{-6}\ \mathrm{C}^{-1}$) and stiffness ($E_{\mathrm{Al2O3}}$ = 385 MPa, E_{WC} = 700 MPa) mismatch (in alumina/WC system reach values exceeded even one gigapascal [3]. The uniformity of distribution of such high stresses in microstructure plays the important role in possibility of material fracture.

Even though, the alumina/tungsten carbide composite system belong to the materials with strong interphase boundary [4] what favors fracture toughness increase, the presence of significant amount of big grains in the microstructure spread this uniform stress distribution and caused reliability decrease.

4 Summary

Measurements of mechanical properties values of investigated materials showed that hardness, as it should be predicted, depended on "the rule of mixtures". Incorporation of the harder carbide phase into the matrix increased the hardness of the material and level of improvement was practically independent on inclusions distribution.

Also fracture toughness of the composites were increased in relation to alumina matrix, to the similar level in the case of both composites. However, it is not obvious why this property was not sensitive on observed microstructure differences. The values of the K_{Ic} coefficient for both composites were similar.

Presented results showed that introduction of tungsten carbide particles into alumina matrix decreased the mean bending strength of the material of about 20 %. But if inclusions were relatively small and uniformly distributed, the reliability of such composite systems raised more than twice when compared to the alumina matrix.

The presence in the microstructure of bigger grains and non uniformity of their size distribution gave the material with similar mean strength but very poor reliability.

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