

## The Effect of Grain Boundaries on the Elastic, Acoustical and Thermo-Physical Properties of Metal-Ceramics Composites

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### Abstract

The purpose of this work was searching of the formation of grain boundaries in metal-ceramics composites at various metal concentrations and sintering temperatures, influence of these boundaries on elastic moduli, coefficient absorption ultrasonic waves (USW) and thermo-conductivity to find the coupling of these properties and to estimate the optimal value of the metal concentration for achieve high quality of ready composites "corundum-stainless steel". These boundaries are formed in the sintering process. In this work, cermets sintered in a high vacuum at different temperatures are investigated. Cermets (metal-ceramic composites) are modern construction materials used in different branches of industry. Their toughness and heat resistance are determined by their elastic and thermo-physical properties. In addition, these properties are significantly dependent on the grain boundaries in the material. The elastic moduli and absorption coefficient were measured by the ultrasonic method at room temperature; measurement of the thermal conductivity coefficient was carried out at temperature 200°C. In addition the samples structure was investigated by optical and scanning electron microscopy (SEM), cermets composition was determined by energy-dispersive X-ray spectroscopy method (EDS). We found two extremes for the concentration dependence of the elastic moduli (E and G) on the stainless steel concentrations, the nature of which is unknown. Similar dependence is observed also for the thermal conductivity coefficient and coefficient absorption ultrasonic waves. A discussion of the results is based on the structure cermet model as multiphase micro heterogeneous media with isotropic physical properties is also presented.

**Keywords:** Metal-ceramic composite; Sintering; Grain boundaries; Elastic and thermo-physical properties

### Introduction

Composite materials, based on ceramics and metals, are widely used in different branches of industry. These materials are often created for the production of constructions with the need for high strength, thermal stability, thermal shock resistance and resistance to aggressive media. Investigations of the physical and technical properties of these materials have been published in periodical issues and monographs [1,2]. Thermal shock resistant composites have to possess high thermal conductivity and mechanical stability, which are determined by the elastic moduli. Thus it is supposed that the composite have to possess these properties simultaneously. However ceramics have small thermo conductivity and high stability while metal possess high thermo conductivity and low stability. Obviously that the mixing of row metal and ceramic powders gives so-called "mechanical mixture" possessed low stability even if the powder mix was compacted by pressure. In this connection to prepare composites usually the sintering process uses. The sintering temperatures can be higher or lower than metal melt temperature. This process leads to activation of the thermo-chemical reactions on grain boundary. The new phase of composite is resulting of sintering process, moreover just her properties determinate the stability and thermo conductivity of ready composite. As of now, there is no uniform understanding about the connection between the elastic and thermo-physical properties of sintered dispersed materials [3].

The purpose of this work was searching of the formation of grain boundaries in metal-ceramics composites at various metal concentrations and sintering temperatures; influence these boundaries on elastic moduli and thermo conductivity to find the coupling of these properties, to estimate the optimal value of the metal concentration for achieve high quality of ready composites "corundum-stainless steel". The present report is development of the authors preceding work complimented by new investigations of cermets microstructure

and their acoustical properties based on elastic moduli and thermo conductivity.

### Materials and Methods

We investigated cermet samples based on  $\alpha\text{-Al}_2\text{O}_3$  in combination with commercial quality stainless steel (18 wt.% Cr, 9 wt.% Ni, 1 wt.% Ti and 72 wt.% Fe). To fabricate a cermet, an initial fine grained mixture was prepared by milling  $\alpha\text{-Al}_2\text{O}_3$  powder (2-25  $\mu\text{m}$ ) in a ball mill in the presence of balls of 1-2 cm in diameter made from stainless steel. The milling was terminated when the steel content in the  $\alpha\text{-Al}_2\text{O}_3$  powder became equal to 2.2, 4.0, 5.5, 11.0 and 21.0 vol%. Then, the mixture obtained was doped with a plasticiser and subjected to dry compaction under a pressure of 100 MPa, followed by sintering in a high vacuum at either 1500 or 1600°C. Finally, the samples were cooled in a furnace at an average rate of 100°C/h and no further treatment was made. The prepared cermets ranged in volume porosity from 3 to 7% and had steel contents from 2.2 to 21.0 vol%. The cermet samples (two series for two temperatures of sintering) were then cut into smaller parts of the required dimensions (10 mm in diameter and 8-15 mm in length), which were further ground and polished, depending on the measuring method. To investigate the elastic properties of the cermets, we measured their density using the hydrostatic method and the velocity of longitudinal and transverse ultrasound waves with a frequency of

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2 MHz. The measurement of mechanical stability requires special preparation of many samples to provide reliable results, whereas for the measurement of dynamic elastic moduli, only one sample is required. Using the pulsed phase-interferometer method [4] made it possible to determine the velocity of the ultrasound waves within an accuracy of 0.1-0.2% and 5% for the elastic moduli. Using the data obtained, we calculated Young's modulus, E, and the shear modulus, G, from the well-known formulas of the elasticity theory for an isotropic medium [4]. Absorption coefficient was measured at 5 MHz frequency of USW within accuracy ~10%. The thermal conductivity coefficient  $\lambda$  was measured using an adiabatic calorimeter, IT- $\lambda$ -400 (Thermometer Company, Russia). The measuring of  $\lambda$  was carried out by a method of comparison with the standard sample. The measurement accuracy of  $\lambda$  was 2-3% for all samples. Examination of the surface structures (cleaved facet) of the cermet samples was made with a JSM-840 scanning electron microscope (JEOL Ltd. Company, Japan), the distribution of particles with size be measured by Mastersizer 3000 (Malvern Instruments Ltd, UK), metallographic and X-ray spectroscopy (EDS) analysis by Thixomet. MACRO (Thixomet, Russia).

## Results and Discussion

In Figure 1, a typical microstructure of the studied cermets, obtained by scanning electron microscopy (SEM), is presented. It can be seen there are three phases in the samples, corundum grains, metal drops and pores. The metal drops have sizes of 2-3  $\mu\text{m}$  and are disposed usually on the joints of the corundum grains, smaller drops

are disposed on the grain faces. It should be noted that inter phase and inter grain boundaries are not observed and the size of the corundum grains did not change after sintering.

Figure 2 shows the curve of the distribution of corundum particles with size in initial powder. From the curve it follows that on the whole the particles have average size ~1.5-2.0  $\mu\text{m}$ . More large particles (10-20  $\mu\text{m}$ ) also are presented. Comparison this between with curve and calculation of the distribution of corundum particles in Figure 3 showed good accordance. Hence at sintering the recrystallization of corundum particles don't observed. Pore and steel particles are distributed of the cermet volume evenly.

From Figures 1-5 it is clear that the grain boundaries phase (probably spinel  $\text{FeAl}_2\text{O}_4$  [5-9]) are invisible. Thus it can suppose that grain boundary phase in cermets is thin layer equal some nm. If the size of this phase is considerably more she became visible. It can see from Figure 6 (new phase cluster size equals 10-20  $\mu\text{m}$ ).

Minimum of absorption coefficient  $\alpha$  can be seen in Figure 7 at steel concentration ~5 vol%. Earlier in the work [6] concentrations dependences of elastic moduli and thermal conductivity coefficient of the same cermets were investigated (Figures 8 and 9). It can be seen that these dependences have minima also at ~5 vol%. Detailed explanation of this effect was presented in work [6]. Briefly it caused by kinetics of grain boundaries formation: she stabilized at this concentration. It should be noted that observed connection  $\alpha$  and  $\lambda$  is describe in acoustical physics of solid state [3,4] as thermo-elastic effect (where

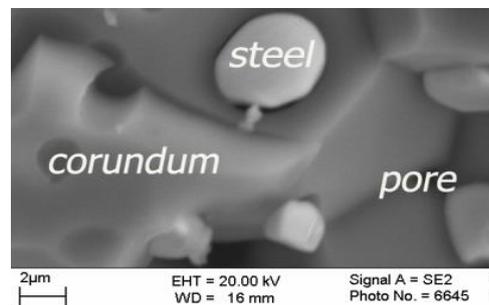


Figure 1: Microstructure of the cermets sample N1 sintered in vacuum at 1500°C by SEM method. It is clear that the grain boundaries are no visible.

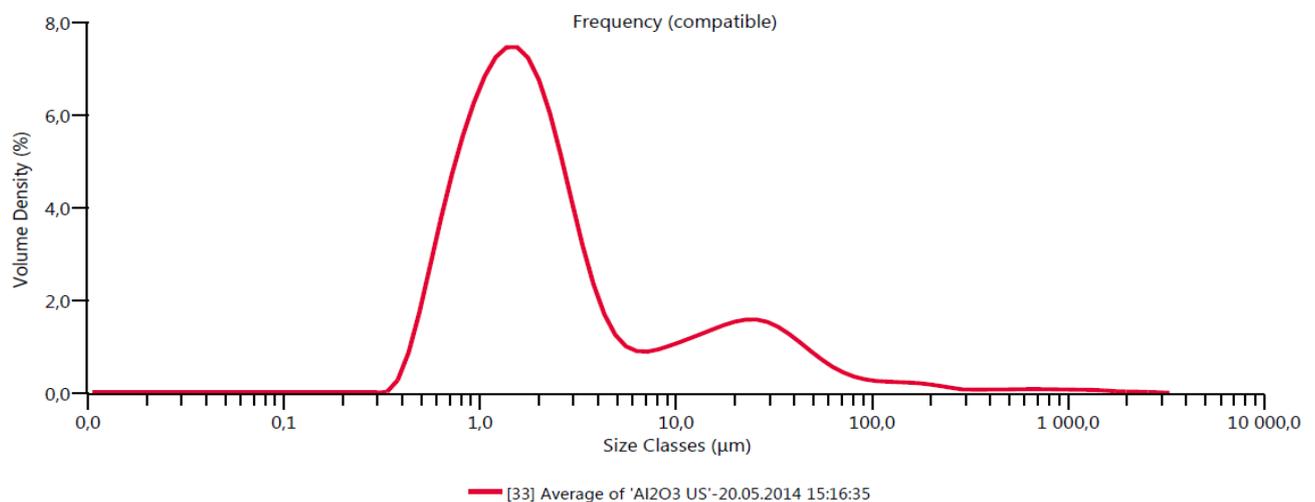


Figure 2: The distribution of corundum particles with size in initial corundum powder.

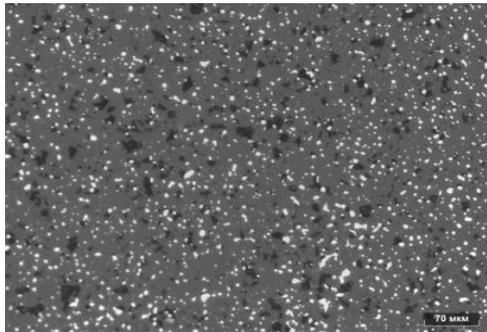


Figure 3: Optical image of cermet N1 shows different cermet areas: black-pores, white-steel, grey-corundum.

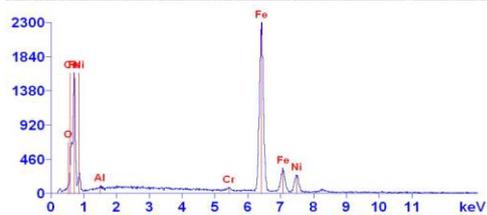
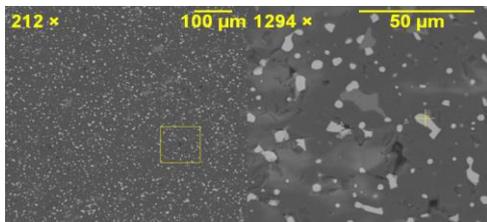


Figure 4: Optical image of cermet N1 (different scale, top) and its corresponding EDS spectrum showing inclusion of steel (bottom).

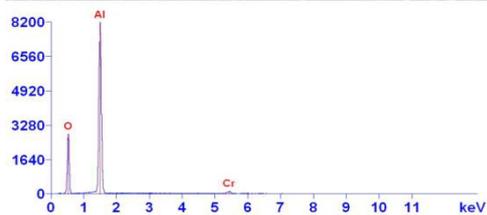
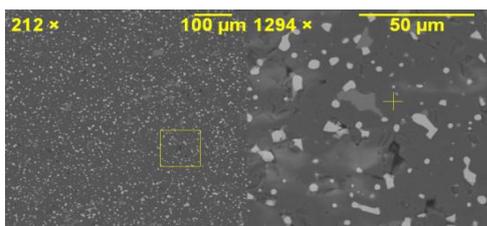


Figure 5: Optical image of cermet N1 (different scale, top) and its corresponding EDS spectrum showing corundum matrix  $Al_2O_3$  (bottom).

$\alpha \sim \lambda$ ). Thus it is believed that the structure of sintered composite is similar to structure of polycrystalline solid body [7-10].

### Conclusion

In the oxide cermets the concentration dependences of elastic moduli, absorption coefficient of USW and the thermal conductivity coefficient have been obtained and discussed; relation between of

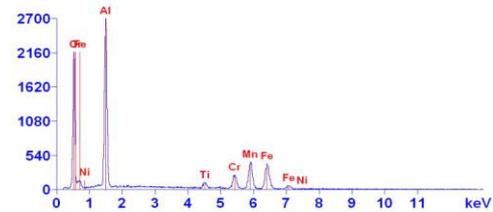
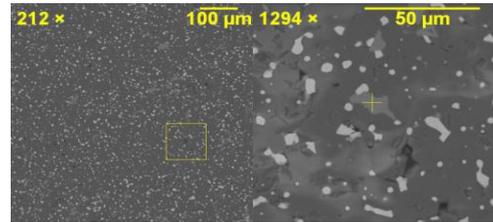


Figure 6: Optical image of cermet N1 (different scale, top) and its corresponding EDS spectrum showing inclusion of new grain boundaries phase made possible spinel  $FeAl_2O_4$  (bottom).

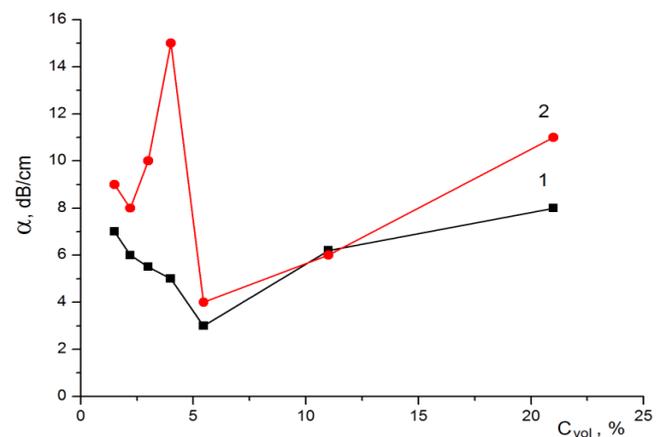


Figure 7: Concentration dependences of the absorption coefficient USW ( $\alpha$ ) in cermets N1, N2 sintered at temperatures of 1500°C (curve 1) and 1600°C (curve 2), respectively.

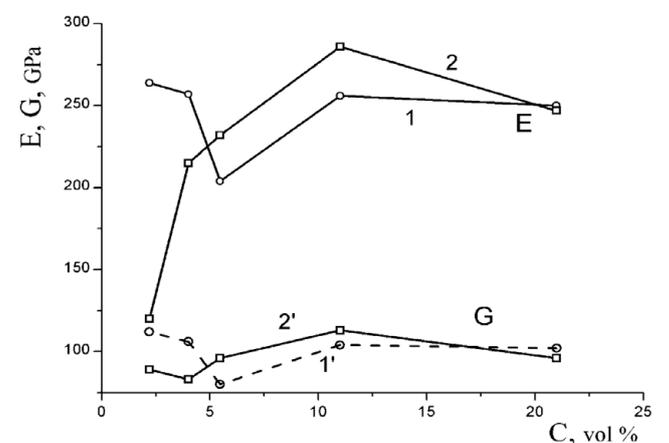
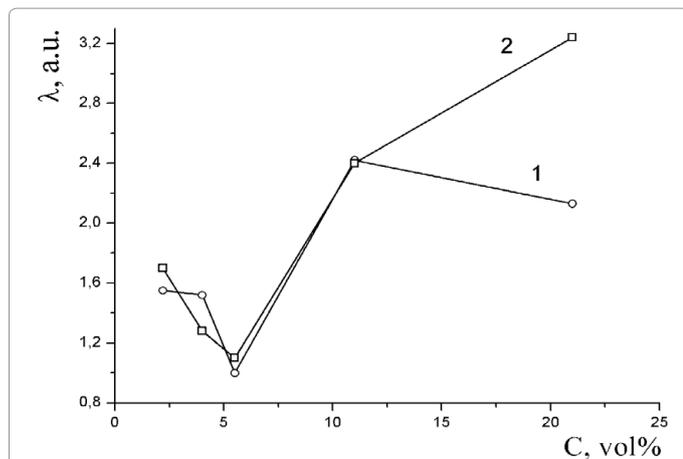


Figure 8: Concentration dependences of elastic moduli, E and G, for cermets, sintered at various temperatures: 1500°C – curves 1 and, 1' and; 1600°C – curves 2 and, 2' [6].



**Figure 9:** Concentration dependences of the thermal conductivity coefficient (arbitrary units) in cermets sintered at temperatures of 1500 (1) and 1600°C (2),  $\lambda=1$  corresponds to the standard sample value [6].

elastic moduli, absorption coefficient and the thermal conductivity for metal concentrations 2-11 vol.% was established; the optimal value of metal concentration for investigated composites “corundum-stainless steel” were determined. To summarize, this work shows us that the thermo-physical and ultrasonic spectroscopy methods are simple and informative in the technology of synthesis of sintered composites.

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