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The measurement of the adhesion force between ceramic particles and metal matrix in ceramic reinforced-metal matrix composites

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ABSTRACT

This paper presents the method for measurement of the adhesion force and fracture strength of the interface between ceramic particles and metal matrix in ceramic reinforced-metal matrix composites. Three samples with the following Cu to Al_2O_3 ratio (in vol.%) were prepared: $98.0Cu/2.0Al_2O_3$, $95.0Cu/5.0Al_2O_3$ and $90Cu/10Al_2O_3$. Furthermore, microwires which contain a few ceramic particles were produced by means of electro etching. The microwires with clearly exposed interface were tested with use of the microtensile tester. The microwires usually break exactly at the interface between the metal matrix and ceramic particle. The force and the interface area were carefully measured and then the fracture strength of the interface was determined. The strength of the interface between ceramic particle and metal matrix was equal to 59 ± 8 MPa and 59 ± 11 MPa in the case of 2% and 5% Al_2O_3 to Cu ratio, respectively. On the other hand, it was significantly lower (38 ± 5 MPa) for the wires made of composite with 10% Al_2O_3.

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1. Introduction

Metal matrix composites (MMCs) are widely used in many fields of engineering, just to mention aviation and aircraft [1,2], automotive [3] and thermal management [4,5]. These materials provided significantly enhanced properties – like higher strength, stiffness and weight savings – in comparison to conventional monolithic materials. Furthermore, they are attractive due to their cost-effectiveness, isotropic properties, and their ability to be processed using the similar technology as in the case of monolithic materials. A large amount of work has been conducted recently in an effort to characterize the mechanical behaviour of particle reinforced MMCs. Not only new production technologies have been developed [6–9] but also analytical and numerical modelling has been done [10–16]. The latest provides an effective and relatively cheap and fast means of predicting effective properties of the composite (e.g. Young modulus) from the known properties of the constituents and revealing deformation and damage characteristics. For instance, Ganesh and Chawla [17] and Peng et al. [18] used finite element method (FEM) for investigation of the influence of the particle shape on the mechanical behaviour of the MMCs. Furthermore, Qing [13] studied the influence of particle arrangement and interface strengths on the mechanical behaviour of the particle reinforced MMCs. Unfortunately, due to the lack of a sufficient knowledge about the mechanical properties of the interface between constituents, the simulations are not able to predict the real values of the materials parameters.

Therefore, there is a need to develop methods for the experimental evaluation of the adhesion force between the metal matrix and the applied reinforcement, especially in the case of micro- and nanocomposites. Fortunately, micro- and nanotechnologies, which have been developed extremely fast recently [19–24], provide tools capable of measuring forces down to a few pN [25–28]. Over the past few years several techniques for the measurement of the mechanical properties of nanostructures have been developed. Young's modulus and fracture strength of silicon nanowires were investigated by Hoffman et al. [28]. Furthermore, in [27] the Atomic Force Microscope (AFM) was used in order to exert forces on pillars with nanometre dimensions and to measure the fracture strength of materials and interfaces. An interesting approach with a micro-sized notched specimen made of tungsten was described by Wurster et al. [15]. In their work, the nanoindentation of the small beam with a notch, made by means of a focused ion beam, is presented. Truong et al. [29] used double cantilever beam tests to assess the mode I interlaminar fracture toughness. Xu et al. [30] applied a combination of the transverse tensile test

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and the unilaterally coupled FEM to evaluate the interfacial bond strength and stress distribution of titanium matrix composites.

However, an alternative approach is necessary in order to investigate MMCs reinforced by ceramic particles. The shape of the ceramic particles is usually random and their size ranges from a few tens of micrometres down to tens of nanometres. Hence, the purpose of this paper is to describe a method for the adhesion force between ceramic particle and metal matrix measurement. The method uses microwires which contain a few ceramic particles. The microwires usually break exactly at the interface between the metal matrix and ceramic particle. The adhesion force is measured with the microtensile tester of our own production.

2. Experimental

The current investigation involves tension of microwires made of MMCs. Three compositions of powder mixtures with the following Cu to Al_2O_3 ratio (in vol.%) were prepared: $98.0Cu/2.0Al_2O_3$, $95.0Cu/5.0Al_2O_3$ and $90Cu/10Al_2O_3$. In the first group commercial copper powder from Sigma Aldrich, with average grain size of about 10 microns and aluminium oxide, α- form, grain size of about 1 micron were used. They were obtained in a mechanical mixing process, using a planetary ball mill (Pulverisette 6, Fritsch) with tungsten carbide balls (∅ 10 mm). The process was conducted in the protective atmosphere of nitrogen at the rotation speed of 200 rpm and at the time of mixing of 4 h. The ratio of the ball to the powder (BPR) was approximately 5:1. The mixing conditions were chosen after earlier experiments of authors [31,32]. Finally the densification process was conducted in Thermal Technology Astro type press using a hot pressing method in a graphite die. The sintering was performed at temperature 1050 °C for 30 min. The heating rate was 10 °C/min. A 30 MPa pressure was applied after reaching the sintering temperature. After the holding time the samples were cooled down naturally in a furnace to room temperature before their removal. A scanning electron microscope (SEM) images of a surface of the samples are shown in Fig. 1. It is clearly visible that the ceramic particles are of random shape.

Next, small rods were precisely cut from the investigated materials using a precise wire saw. The rods were then put on a special holder and carefully electro etched in the phosphoric acid solution (40%) in order to produce microwires. The sample holder (Fig. 2a) consists of two aluminium bars to which the rode made of investigated composite is clamped by two small screws. The bars are connected to each other by parts made of PCV. Only the rod and the aluminium bars conduct the electricity which allows to easily control the conductivity of a microwire while it is etched from the rod. After the rod had been mounted on the sample holder, its both endings were covered with varnish in order to protect them from etching. Hence, the microwire was created in the middle of the rod. The positive voltage is applied to one side of the holder and then the holder is immersed in the acid solution. On the other hand the cathode was made of stainless steel. After a few minutes of the precise electro etching a microwire with an interface between ceramic particle and copper should be produced. When the diameter of the microwire was lower than approximately 60 μm then after every 20 s the etching was stopped and the microwire was carefully investigated upon the optical microscope and the microwire's resistance was accurately measured. The electro etching was continued until the ceramic particle was clearly visible in optical microscope image and until microwire stopped conducting the electric current. If a microwire does not conduct the electric current then it is assumed that the interface is fully exposed. The microwires were further investigated with SEM. The thickness of the wires and the position of the ceramic particles were precisely evaluated.

Furthermore, the sample holder with a microwire was mounted on the microtensile tester of our own production (Fig. 2b and c). The microtensile tester consists of two stages, to which two endings of the microwire are fixed. In order to make the stage stable and sensitive to very low loads, the first stage is mounted at the end of four thin, flat springs on which 4 strain gauges are glued. The springs are made of steel. Their height is 70 mm, their width is 5 mm and their thickness is 0.2 mm. The strain gauge bridge is used to precisely measure the force which is applied on a microwire. The force measurement module was calibrated using precise

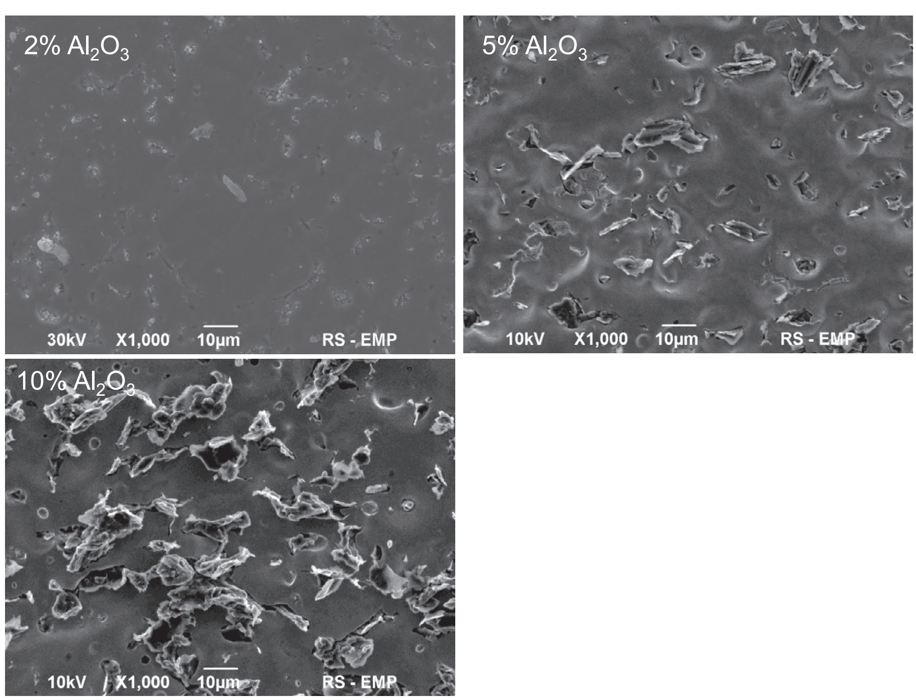

Fig. 1. The SEM image of a surface composites surface. The ceramic particles have usually very complicated and irregular shape.

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