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Reinforcement of Conducting Silver-based Materials

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Silver is a well-known material in the field of contact materials because of its high electrical and thermal conductivity. However, due to its bad mechanical and switching properties, silver alloys or reinforcements of the ductile silver matrix are required. Different reinforcements, e. g. tungsten, tungsten carbide, nickel, cadmium oxide or tin oxide, are used in different sectors of switches. To reach an optimal distribution of these reinforcements, various manufacturing techniques (e. g. powder blending, preform infiltration, wet-chemical methods, internal oxidation) are being used for the production of these contact materials. Each of these manufacturing routes offers different advantages and disadvantages. The mechanical alloying process displays a successful and efficient method to produce particle-reinforced metal-matrix composite powders. This contribution presents the obtained fine disperse microstructure of tungsten-particle-reinforced silver composite powders produced by the mechanical alloying process and displays this technique as possible route to provide feedstock powders for subsequent consolidation processes.

Keywords: mechanical alloying, silver/tungsten composite powder, microstructure, particle-reinforced, conducting materials.

1. INTRODUCTION

1.1. Mechanical Alloying

Koch et al. [1] describes the mechanical alloying as a synthesis of materials by high-energy ball-milling of powders. In order to produce oxide-dispersion-strengthened (ODS) alloys, John Benjamin and its co-workers developed the mechanical alloying process around 1966 [1 – 6].

The central procedure of mechanical alloying is the collision between the balls. The powder between them and between the balls and the chamber wall undergoes deformation, fraction and cold-welding processes. Three different powder combination systems are characteristic for the described process: (i) ductile/ductile, (ii) brittle/brittle or (iii) brittle/ductile [1 – 4, 7, 8].

The mechanical alloying of (i) ductile/ductile particles is characterized by flattening and cold-welding processes to lamellar structures, which form homogeneous equiaxed particles after sufficiently often repeated re-welding and fracturing processes. Further, the fracturing into smaller particles and also the deformation of the system (ii) brittle/brittle is described. Within the system (iii) ductile/brittle, the brittle particles were fractured in the mechanical alloying process and the ductile particles were deformed at the same time. Furthermore, the brittle particles were trapped between the ductile particles. Continuing fracture and cold-welding results in a homogeneous distribution of the brittle component in the ductile matrix [1, 3, 7, 8, 10].

The process variables such as the type of the mill, the milling speed, the milling time, the type and size of the grinding medium, the ball-to-powder-ratio, the milling atmosphere, the temperature of milling and the process control agent are influencing the result of the milling process [3 – 5, 8, 9]. Further, the increase in temperature during the process is mentioned [3].

As an important problem the contamination of the powder during the process is named [1, 2].

Usually approximately 1 wt.-% – 2 wt.-% of a process control agent (PCA) are added to the powder during the milling of the ductile particles. These PCAs, e. g. stearic acid, prevent excessive cold welding between the particles as well as between the particles and the balls or the chamber wall [2].

Various milling equipment is described by [1 – 5, 9]. This high-energy milling equipment differs in the capacity, efficiency and additional arrangements, for example, for cooling or heating processes [3, 4].

1.2. Contact Materials

The electrical contact describes the situation of two devices exposed to each other in a current-carrying way. The devices are labeled as contact pieces. These contacts are subject to different mechanical, electrical and thermal effects as well as environmental influences. Different materials are used in the field of contact materials. Examples are very several variations of platinum, gold, tungsten, copper or silver. This contribution focuses on the investigation of silver-based materials [12 – 14].

Silver is appropriate for applications in switches because of the highest electrical and thermal conductivity. Because of its poor welding and wear resistance as well as its tendency to material migration in direct current applications, silver satisfies the requirements as a contact material only in a limited range of electrical applications. On that account silver alloys or silver composite materials are used to meet the requirements. AgNi 0.15 (fine-grain silver) and AgCu alloys are typical examples for silver alloys. Different reinforcements such as the composite

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material systems silver tungsten (Ag/W), silver tungsten carbide (Ag/WC), silver nickel (Ag/Ni), silver cadmium oxide (Ag/CdO) and silver tin oxide (Ag/SnO_2) are being used in different sectors of switches. To produce these composite materials, different production routes are possible, e. g. precursor infiltration, powder blending, wet-chemical ways and internal oxidation. These routes offer specific advantages and disadvantages [12 – 17].

This contribution focuses on silver/tungsten materials which are applicable for high currents. These contact materials are characterized by high reinforcement contents. To produce these Ag/W-materials different production routes are possible, e. g. precursor infiltration or powder blending. The precursor infiltration offers dense composite materials with an interpenetrating system of the tungsten and silver component. To produce the infiltration materials adequate W-precursors are necessary. Although large powder amounts can be achieved by means of the powder blending way, this method is limited by the distribution of the tungsten-component in the silver-matrix caused by the difference of the reinforcement and matrix particles which are blended. Only an accurate adjustment of the powder fraction of the matrix and reinforcement improve this reinforcement distribution in the matrix [12 – 17].

Caused by the advantages and disadvantages of these routes the objective within this contribution is to display an alternative method to produce feedstock Ag/W powders for subsequent consolidation processes. Therefore, the mechanical alloying process is a promising route to obtain feedstock powder with a fine-disperse reinforcement distribution. This route provides the possibility to incorporate the reinforcement component in the matrix material [7] and to achieve composite powder for subsequent consolidation processes.

2. EXPERIMENTAL DETAILS

Elemental Ag- and W-powders with two different tungsten-particle sizes and contents (1.5 µm and 5 µm, 90 wt.-% and 65 wt.-% W) were pre-mixed and then mechanical alloyed in a Simoloyer® ball mill with ceramic configuration (ceramic-balls, -rotor and -chamber) for different milling times, at argon atmosphere, with a ball-to-powder-ratio of 10 : 1 and with a rotation speed of 400 rpm. These different contents and particle sizes were selected to detect the incorporation of a wide range of the reinforcement content as well as the particle size in the silver matrix. The Simoloyer® (Zoz GmbH, Wenden, Germany) is characterized by a fixed milling chamber and a spinning rotor which causes acceleration to the milling balls and so to the milling powder. It is possible to work continuous at approximately room temperature because of an integrated water cooling system. Furthermore, it is possible to scale up the milling chamber to a semi-industrial volume and so it is possible to produce a semi-industrial powder amount by an up-scalable process [11].

3. RESULTS

3.1. Mechanical Alloying Process

The silver tungsten composite powders with 10 wt.-% and 35 wt.-% silver were observed optically and assessed after different milling times. The composite powders with both silver contents maintain their powder form during the mechanical alloying process. No excessive cold welding of the powder at the milling balls or the chamber wall of the mill can be observed. The addition of process control agents is not necessary. This shows an outstanding advantage to prevent PCA expulsion processes and their disadvantages like pores or contaminations.

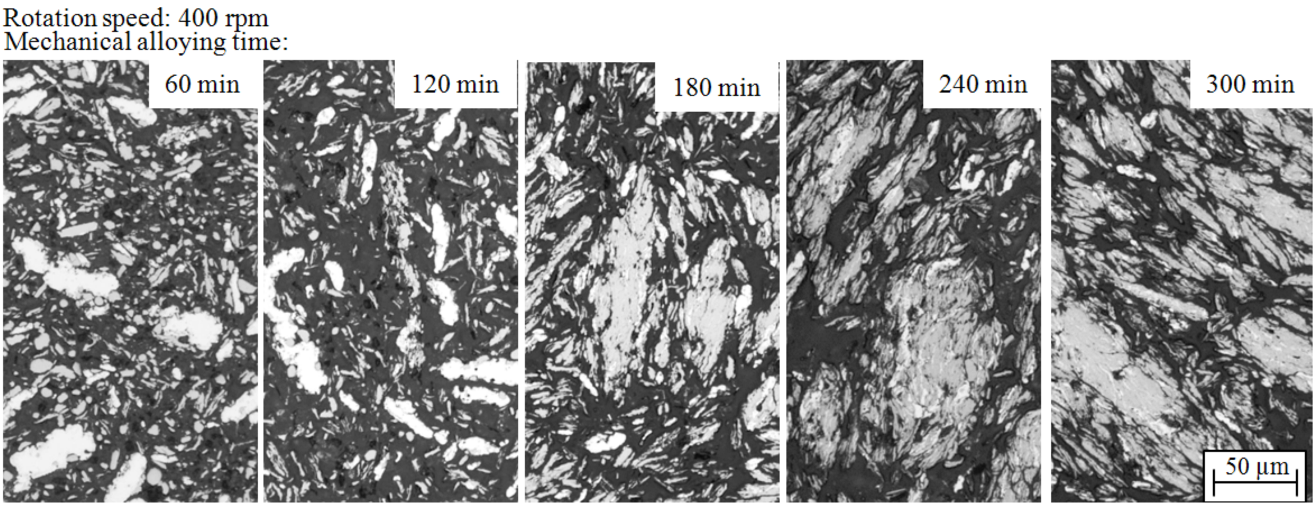
3.2. Microscopically Characterization

After different mechanical alloying times the composite powder was microscopically characterized by means of optical microscopy (OM). Selected samples were investigated by scanning electron microscopy (SEM) and further selected samples by field-emission scanning electron microscopy (FE-SEM). During these investigations the focus was on the fine disperse distribution of the tungsten component in the silver matrix. Cross section polish specimens of the Ag/W composite powders were prepared by the infiltration of the composite powder with an epoxy resin and further cross section preparation steps. For FE-SEM investigations selected samples were prepared in a subsequent step by ion polishing.

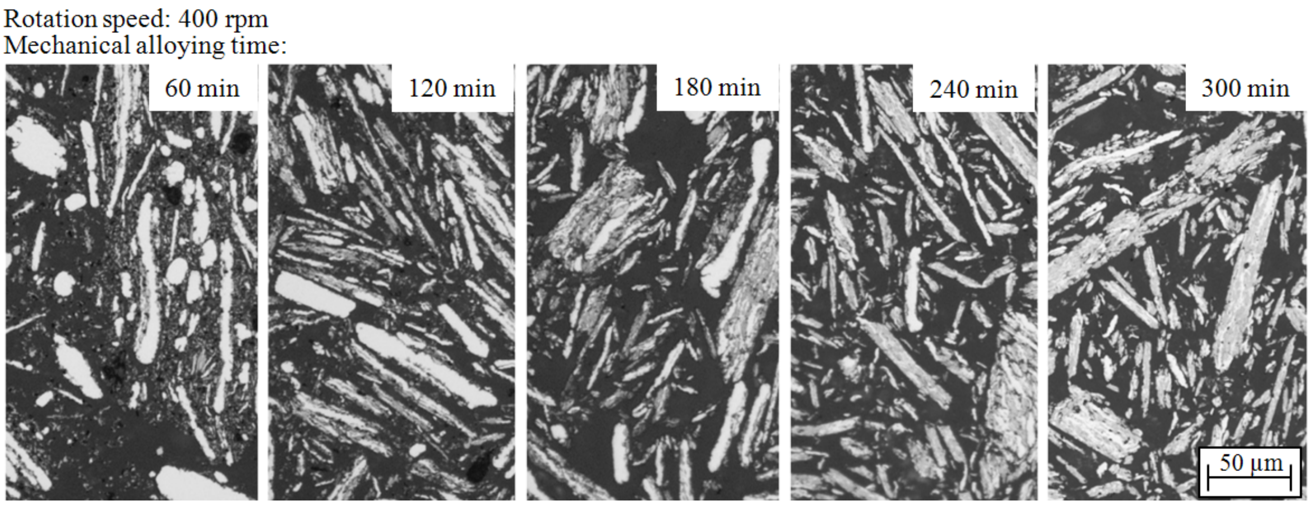
The OM-images in Figure 1, a, b, display the evolution of the composite powder for five different mechanical alloying times (60, 120, 180, 240, and 300 min) for the silver/tungsten composite powder with 10 wt.-% and 35 wt.-% silver. According to the evolution which is described for the system ductile/brittle in Kudashov [7], the silver component was deformed into a typical lamella structure after short processing times. The tungsten component is attached to the ductile silver component, but in contrast to the evolution indicated by Kudashov [7], it is not ground. This component is also subjected to a deformation process, which is known from the system ductile/ductile in the early states, whereby this deformation process starts in a subsequent state of the observed mechanical alloying process.

With repeated deformation, fraction and welding processes during the mechanical alloying the composite powder develops a coarse lamellar structure and after an appropriate mechanical alloying time the composite powder becomes more and more homogeneous. The described process takes place for both silver contents (10 wt.-% and 35 wt.-%) and tungsten particle sizes.

The finished composite powders are characterized by a very homogeneous distribution of a significantly lamellar tungsten component in the silver matrix, which is clearly visible in the atomic number contrast (QBSD-detector) SEM-image in Figure 2 exemplarily for 35 wt.-% silver. More details are displayed for example for the low silver content of 10 wt.-% by means of the InLens-detector image of a FE-SEM in Figure 3. Obviously, a combination of the two well known systems ductile/ductile and ductile/brittle exists for the material combination silver/tungsten. Thereby, the silver component displays a much higher ductility than the tungsten component. The mechanical alloying energy is high enough to deform both components significantly and to obtain a fine disperse distribution of the more brittle component within the more ductile component.



a



b

Fig. 1. Silver/tungsten composite powder with 10 wt.-% (a) and 35 wt.-% (b) silver after different mechanical alloying times 60, 120, 180, 240 and 300 min (powder section, OM)

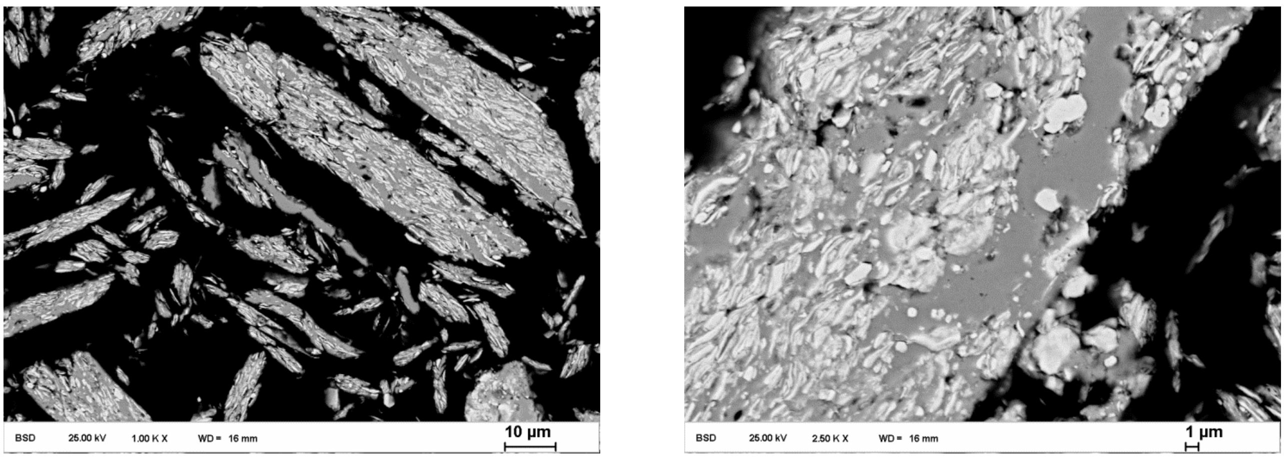


Fig. 2. Silver/tungsten composite powder with 35 wt.-% silver after 300 min mechanical alloying in an overview (left) and in detail (right), (powder section, SEM, QBSD-detector)

It is also visible, that the mechanical alloying process is a statistical process. After a defined time, only a part of the powder undergoes the central procedure of deformation, fraction and cold-welding of the mechanical alloying process and after an appropriate time, statistically the whole powder undergoes the process. So it is possible that a part of the powder undergoes more fraction, deformation and cold welding processes than another part.

This statistically distributed effect is made evident for example by the silver powder after 60 min as shown in Figure 1, b, and by a higher deformation grade of some particles of the tungsten component in Figure 2. Especially

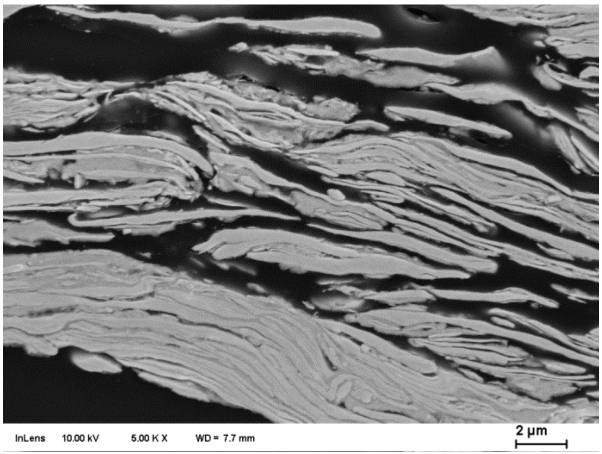


Fig. 3. Silver/tungsten composite powder with 10 wt.-% silver after 300 min mechanical alloying in detail (powder section after ion polishing, field-emission SEM, InLens-Detector).

the fine lamellar structure, exemplarily seen in Figure 3 for tungsten/silver composite powder with 10 wt.-% silver, displays an intensive binding between the fine silver sub lamellas and the tungsten lamellas. The more the lamella structure is formed and the central procedures deformation, fraction and cold-welding of the mechanical alloying process takes place, the more intensive becomes the binding of the components. Besides the formation of the lamella structure as well as the composite powder formation is significantly influenced by the tungsten particle size and the silver content.

3.3. X-ray diffractometry

Selected composite powders were characterized by the X-ray diffractometry. The phase analysis as a part of the X-ray diffractometry investigations displays only the phases silver and tungsten in the silver/tungsten composite powder after different milling times. Depending on the detection limit of the X-ray diffractometry no other phase was detected. According to that no phase formation during the mechanical alloying process takes place. Only a fine disperse distribution of the tungsten component in the silver matrix with an intensive binding of the components can be observed. Furthermore, no significant contamination can be detected after the different mechanical alloying times. So this main drawback of the mechanical alloying process can be invalidated.

Moreover, a significant peak widening is shown in the affiliated diffractograms with rising mechanical alloying time, as shown in Figure 4 exemplarily for the composite powder with 10 wt.-% silver. The quantification of this peak widening shows a significant decrease of the crystallite size of silver as well as of tungsten with rising milling time. The crystallite size development of the tungsten particles displays a much more significant decrease during the first 150 min of the mechanical alloying process than between 150 min and 300 min. The decrease of the silver crystallite size is also highly significant but much more continuous in comparison to the tungsten crystallite size. But the decrease within the second mechanical alloying period between 150 min and 300 min is also lower.

4. CONCLUSIONS AND PERSPECTIVE

The investigations have shown that the mechanical alloying process is a suitable and efficient technology to produce silver/tungsten composite powders in a wide range of the silver content as feedstock for subsequent consolidation processes. The composite powders with 10 wt.-% and 35 wt.-% silver show a homogeneous distribution of the tungsten component after an appropriate mechanical alloying time of approximately 300 min. Beside the

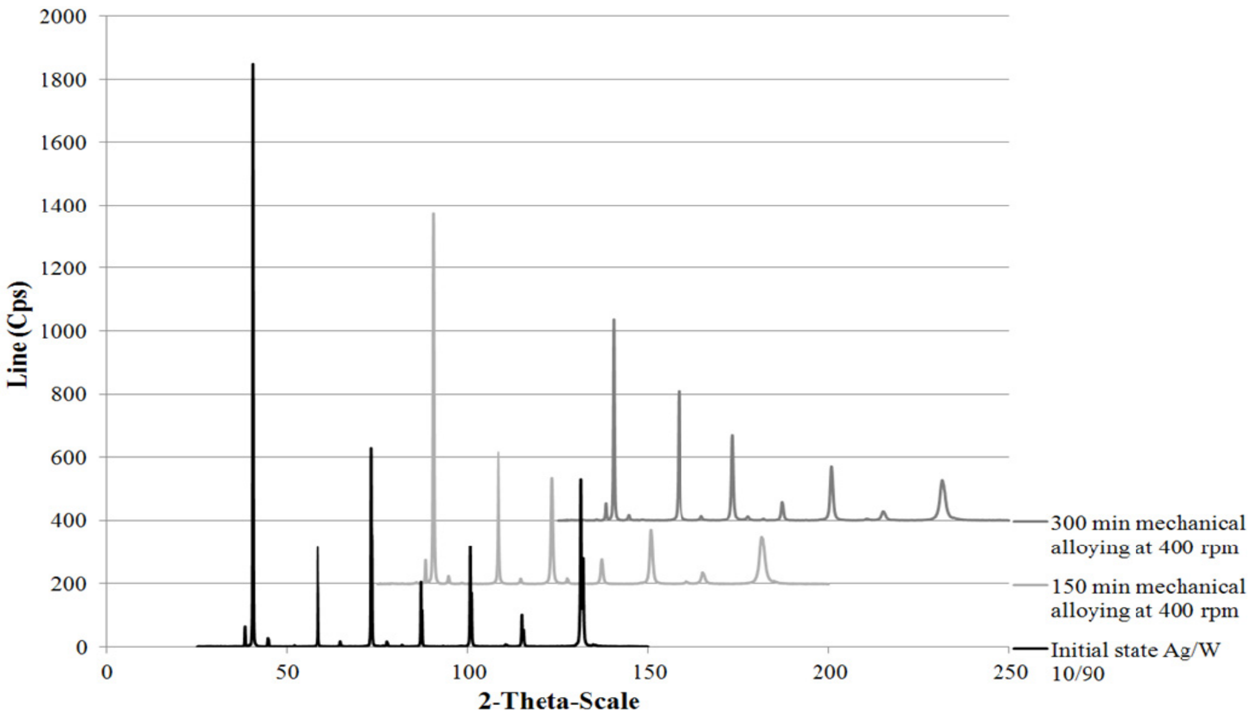


Fig. 4. X-ray diffractograms of silver/tungsten composite powder with 10 wt.-% silver at the initial state and after 150 min and 300 min mechanical alloying.

successful preparation of the composite powders further positive aspects can be named. During the process no process control agents are necessary. Due to the combination of the systems ductile/ductile and ductile/brittle it is possible to detect a statistical distribution of the mechanical alloying process in the powder. Consequently, an analogy between the successful incorporation of the tungsten component in the silver matrix and the deformation ratio of the tungsten component can be detected. Furthermore, no significant contaminations can be clarified. Therefore the two well known disadvantages of the mechanical alloying process, the contamination during the process as well as the usage of PCAs can be avoided. Current investigations deal with the consolidation of the composite powders to compact materials. Moreover it is planned to analyse the switching properties of selected materials.

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