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## PROGRESS IN FABRICATION TECHNOLOGY OF SILVER-BASED CONTACT MATERIALS WITH PARTICULAR ACCOUNT OF THE Ag-Re AND Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub> COMPOSITES

### POSTĘP W TECHNOLOGII WYTWARZANIA MATERIAŁÓW STYKOWYCH NA BAZIE SREBRA ZE SZCZEGÓLNYM UWZGLĘDNIENIEM KOMPOZYTÓW Ag-Re I Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub>

The paper outlines technologies currently used for the production of the Ag-Re10 and Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub> contact materials in a form of wires and solid and bimetallic rivets. Their characteristic parameters, including physical and mechanical properties and microstructure, are given. It has been found that the level of these parameters, particularly electrical properties (resistance to electric arc erosion), is unsatisfactory considering the present requirements, which applies mainly to the new Ag-Re10 [wt%] alloy, so far not fully technologically mastered. Therefore, under this work a new method for the production of this type of materials has been designed and the related research works were undertaken. The new-generation contact materials in a form of nanostructured composites will be characterised by similar chemical compositions as those specified above but with increased functional properties, including enhanced resistance to arc erosion. In this paper preliminary results of the examination of structure and properties of semi-products obtained by new technology based on powder metallurgy techniques are presented. Conditions for pressure consolidation and plastic consolidation applied for material processing into wires and rivets (solid and bimetallic) have been determined.

*Keywords:* electric contacts, nanocomposite, contact materials, electrical properties, tested methods, arc erosion

W referacie przedstawiono stosowany aktualnie zarys technologii wytwarzania materiałów stykowych Ag-Re10 i Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub> w postaci drutów oraz nitów litych i bimetalowych i jednocześnie podano ich charakterystyczne parametry obejmujące właściwości fizyko-mechaniczne i mikrostrukturę. Stwierdzono, że poziom uzyskiwanych właściwości szczególnie elektrycznych (odporność na erozję łuku elektrycznego) jest niezadawalający w świetle aktualnych wymagań, co dotyczy głównie nowego jeszcze nie w pełni opanowanego technologicznie materiału Ag-Re10% wag. Zaprojektowano więc nowy sposób wytwarzania tego typu materiałów i podjęto prace badawczo rozwojowe w tym zakresie. Ich celem jest opracowanie sposobu wytwarzania nowej generacji materiałów stykowych w postaci kompozytów o podobnym składzie chemicznym jednakże charakteryzujących się nanostrukturą i znacznie wyższymi parametrami – głównie odpornością na działanie łuku elektrycznego. Przedstawiono wstępne wyniki badań obejmujące syntezę mechaniczną oraz określono warunki konsolidacji odkształceniowej i plastycznej w procesie ich przeróbki na druty i nity lite i bimetalowe, bazując na procesach z obszaru metalurgii proszków.

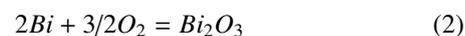
#### 1. Introduction

The contact materials produced at present at the INMET Metals Processing Plant of the Institute of Non-Ferrous Metals (INMET-IMN) are listed in Table 1.

The Ag-Re10 contact material is still at the stage of development of its optimal production technology, and therefore, it has not been included into Table 1, which lists only commercially available products.

Technology for the production of electrical contacts from the Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub> composite material used at present at INMET-IMN has been developed at our Institute and registered at the Polish Patent Office [1, 2]. The method developed is based on fabrication of the alloy powder AgSn7.5Bi0.35 using water atomiser operating at a maximum pressure of

200 bars. The powder obtained has favourable grain size distribution of 40-150 μm, and after mechanical mixing it is subjected to consolidation into compacts about 90 mm in diameter and about 300 mm long. This process is followed by an internal oxidation aimed at separating silver, tin and bismuth in a form of fine particles uniformly distributed within the Ag matrix. Internal oxidation is conducted in the atmosphere of pure oxygen, and the measure of oxidation of Sn and Bi is an increase in mass of the compacts, which should be at least 1.95% in accordance with stoichiometry of oxidation reactions for both elements, i.e. (1) and (2):



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TABLE 1  
Grades of contact materials currently produced at INMET-IMN

No	Material	Wire diameter mm	R <sub>m</sub> MPa	A <sub>100</sub> %	HV <sub>0,2</sub>
1	AgCdO10	2,9	247	31	76
2	AgCdO13,5	2,9	255	29	79
3	AgSnO <sub>2</sub> Bi <sub>2</sub> O <sub>3</sub> 10	2,9	295	18	97
4	AgSn <sub>2</sub> In <sub>2</sub> O <sub>3</sub> 12	2,9	315	12	95
5	AgNi10	2,9	230	30	75
6	AgFe10	2,9	220	35	62
7	AgNi015	2,9	200	35	40
8	Ag-MM*	2,9	300	17	100

\*- 1%wt. of rare earth metals in which there are: 50%wt cerium (Ce), 30% wt lanthanum (La), 10% wt. neodymium (Nd) and the rest is the other rare earth elements

The oxidised material is then subjected to hot compacting at 800°C for about 45 seconds using hydraulic press with a maximum capacity of 800 ton. Next, the compacts are subjected to an additional operation of sintering for 3 hours at the temperature of about 930°C in air atmosphere, followed by multi-strand extrusion (4 strands, each 4 mm in diameter) conducted by means of 800 ton hydraulic press. The wire obtained was then subjected to drawing to a final diameter so as to be suitable for the production of solid and bimetallic contact rivets using two-stroke cold upsetting machines. Microstructure of the products obtained by this method is illustrated in Fig. 1, 2 and 3. Exemplary results of tests carried out at the Institute/Department of Electrical Apparatus of the Lodz University of Technology [9-10] which were intended to determine contacts resistance to an electric arc erosion in case of the Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub> contacts and their other grades produced at INMET IMN, are presented in Fig. 4.

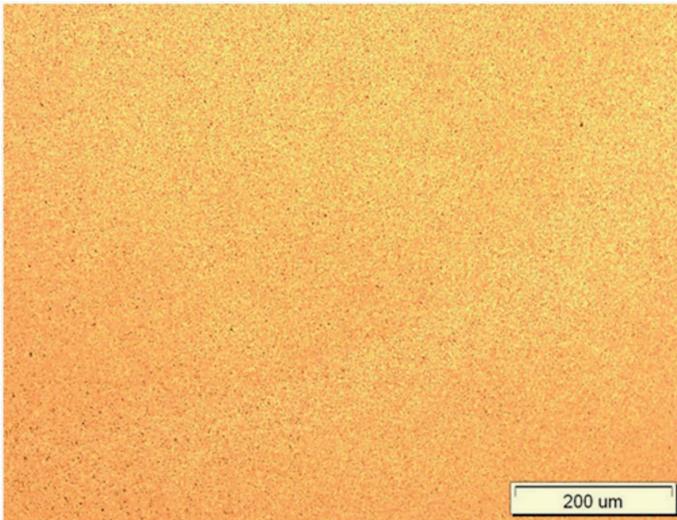


Fig. 1. Microstructure of the Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub> wire on its cross-section

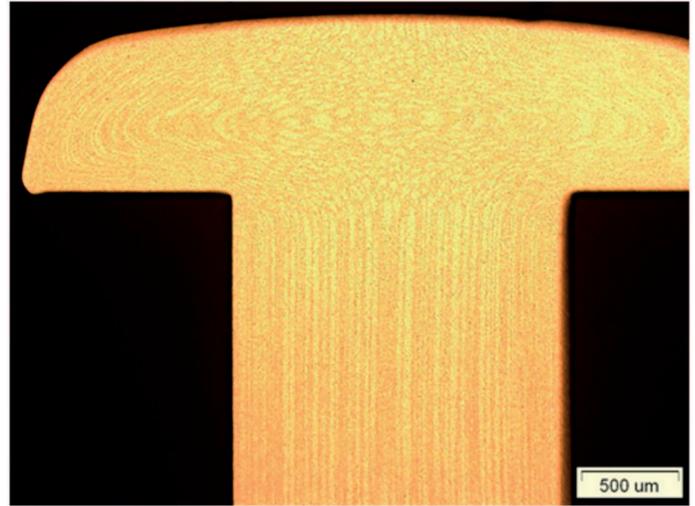


Fig. 2. Microstructure of the Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub>, solid contact rivet (longitudinal section)

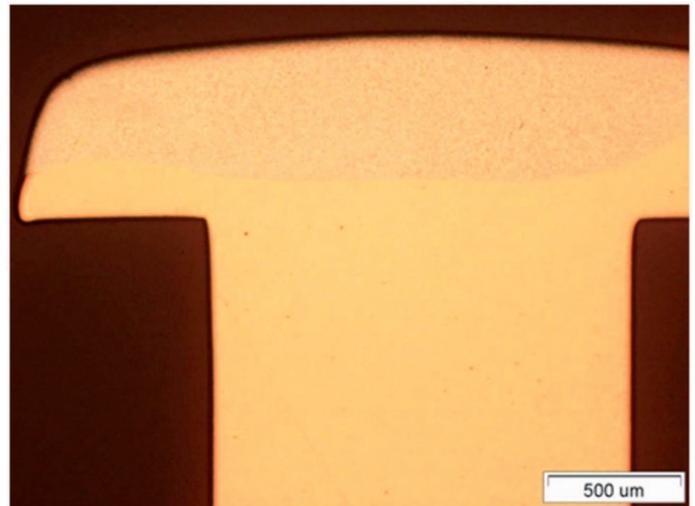


Fig. 3. Microstructure of the Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub>, bimetallic contact rivet (longitudinal section)

The new product, for which preliminary technological guidelines for the production of solid and bimetallic contact rivets have been developed, is the Ag-Re10 [wt%] contact material [4]. These guidelines make it possible to start pilot-scale production of this type of composite contact materials based of powder metallurgy processes. The starting materials are silver powders (min. 99.98% Ag in purity) with a grain size of 1 – 90 μm, and rhenium powders 99.8 wt% in purity and below 5 μm in grain size. Tests were made with mixing these powders followed by their consolidation by means of isostatic press. The compacts obtained by this method, about 18 mm in diameter, were then subjected to hot plastic working with the use of horizontal hydraulic press operating under the KOBO system. The semi-product obtained, i.e. wire 4 mm in diameter, was subjected to drawing to the final diameters of 2.90 mm and 1.90 mm, and used to fabricate bimetallic contact rivets shown in Fig. 5.

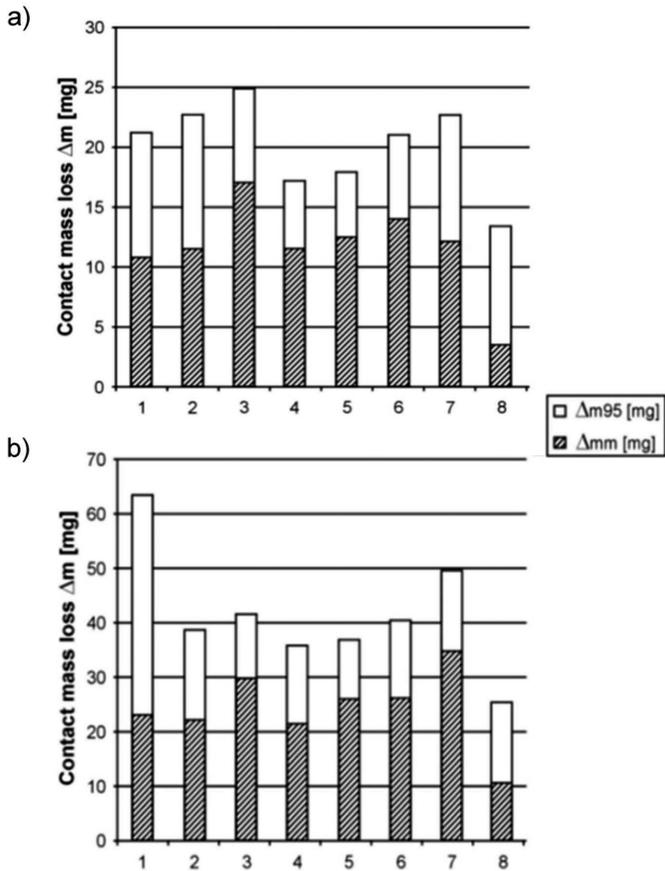


Fig. 4. Results from tests for contact rivets' resistance to electric arc erosion. No. of switching cycles: a) 50 000; b) 100 000. The grades of contact materials under tests: 1 – AgCdO10; 2 – AgCdO13.5; 3 – AgSnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub>; 4 – AgSnO<sub>2</sub>In<sub>2</sub>O<sub>3</sub>; 5 – AgNi015; 6 – AgNi10; 7 – AgFe10; 8 – Ag-MM



Fig. 5. Bimetallic contact rivets made from the Ag-Re10 contact material

The produced rivets were subjected to arc erosion resistance test using model device for testing this parameter. The obtained initial results of measurement of contacts mass loss in function of switching amounts (max.  $50 \times 10^3$ ) and current voltage of 10A are presented in Fig. 6.

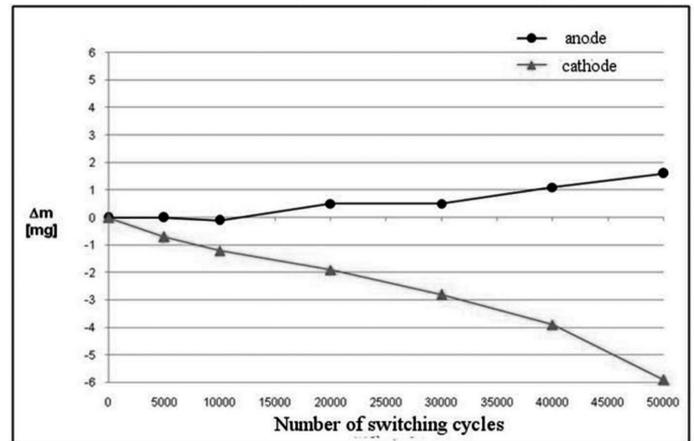


Fig. 6. The Ag-Re10 contact mass loss in dependence on a number of switching cycles

The results of preliminary tests related to fabrication technology and quality of the new Ag-Re10 contact material were found to be unsatisfactory and, therefore, it was decided to undertake further research on this material [5, 6] in order to develop technology for its production in a form of nanocomposite material. The main requirements to be met by this new material were enhanced physical and mechanical properties, particularly higher resistance to arc erosion. To the scope of the initiated new research project the Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub> contact material was included because it has been produced at INMET-IMN for over 15 years, it exhibits very good physical, mechanical and functional properties, and it was expected to considerably enhance these properties by producing it in a nanocomposite form.

## 2. Preliminary results of fabrication of the Ag-Re10 nano-composite material

### 2.1. Mechanical Synthesis of a Mixture of Powders of Silver and Rhenium

Silver and rhenium are the metals differing considerably in physical and chemical properties, particularly such as melting temperature (Ag = 961°C, Re = 3180°C), hardness (Ag = 80HV, Re = 250HV), and resistivity (Ag = 0,0163Ω mm<sup>2</sup>/m, Re = 0,21Ω mm<sup>2</sup>/m). Their solid-state solubility and liquid miscibility are practically equal to zero. Despite that, rhenium is being added to some contact materials such as AgFe10Re0.4 and Ag-W50Re because it has been found that this addition improves performance of these materials [7, 8]. In case of the designed by us silver-rhenium composite material containing as much as 10 wt% Re it was decided to employ the process of mechanical synthesis in order to obtain an alloy of a nanocrystalline structure. For that reason an experimental high-energy ball mill was built (Fig. 7) and used in the tests of mechanical alloying (in argon atmosphere) and microstructure refinement by deformation of the powders being milled, which applies particularly to silver. Chemical composition, grain size distribution and other physical parameters of the powders are specified in Tables 2 and 3.

Chemical composition of the powders of silver and rhenium used in mechanical synthesis process

Metal analysed	Ag	Re	C	W	Fe	Cu	Ni	Cr	Pb	Zn
	% wt									
Ag	The rest	-	-	-	0,0048	0,0038	-	-	<0,0002	<0,0002
Re	-	The rest	0,11	0,67	0,014	-	0,0017	0,0005	-	-

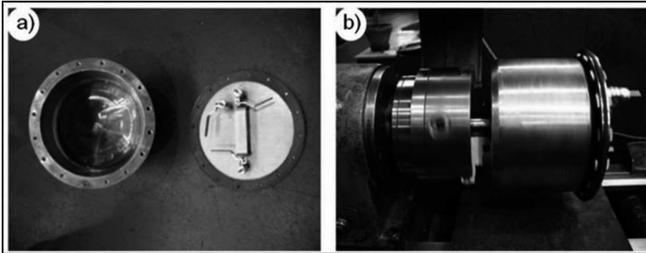


Fig. 7. Equipment for conducting mechanical synthesis and microstructure refining in high-energy ball mill: (a) inside of a drum, (b) mounted drum

TABLE 3

Physical and technological parameters of the powders of silver and rhenium used in mechanical synthesis process

Metallic element	Density, g/cm <sup>3</sup>	Specific surface area, m <sup>2</sup> /g	Technological indices on size of particles, μm		
			d10	d50	d90
Ag	10,40	0,06	8	19	38
Re	18,34	1,58	1	2	4

Morphology of both grades of powders has also been examined, the results being shown in Fig. 8.

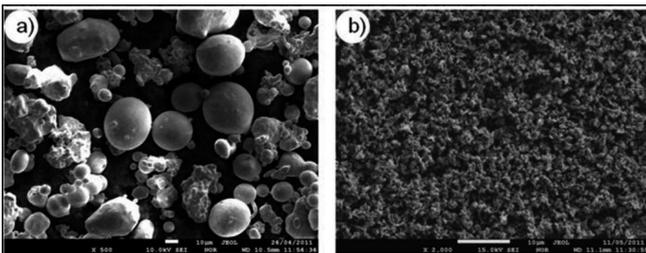


Fig. 8. Morphology of the powders of silver and rhenium used for fabrication of the Ag-Re10 nanocomposite: (a) silver, (b) rhenium

Efficiency of the process of mechanical synthesis of the Ag-Re10 nanocomposite was assessed taking a sample obtained by mechanical mixing of the powders of Ag and Re as a reference. This mixed powder is shown in Fig. 9.

The process of mechanical synthesis was conducted for about 150 hours. After 10, 20, 31, 55, 87, 118 and 150 hours of milling samples were taken in order to examine process efficiency. This examination included X-ray phase analysis, measurements of a size of crystallites of silver and rhenium, and the assessment of morphology of the powders under milling. Selected results from this examination are shown in Fig. 10-13.

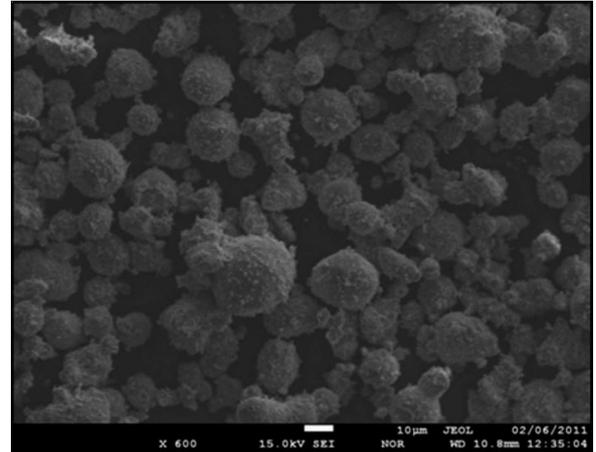


Fig. 9. A mixture of the powders of silver and rhenium as a reference material for the process of mechanical synthesis of these powders

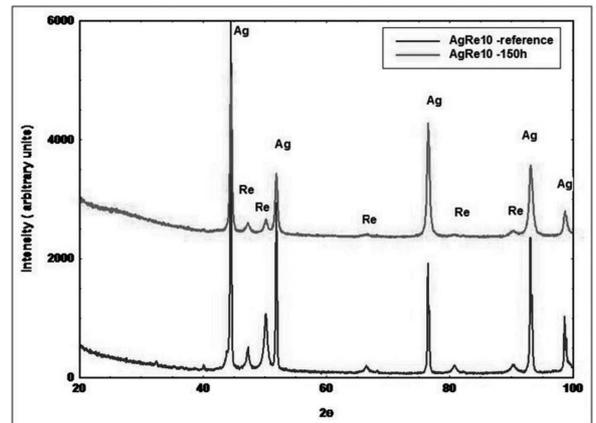


Fig. 10. The X-ray diffraction pattern of the AgRe10 powder mixture before and after 150 hours of its milling in high-energy ball mill

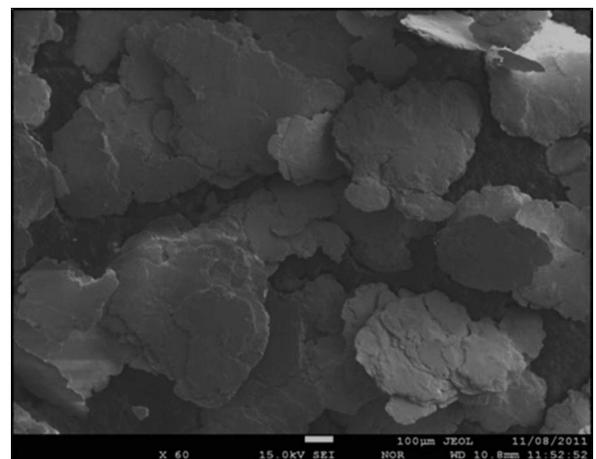


Fig. 11. Morphology of the AgRe10 powder after 150 hours of milling

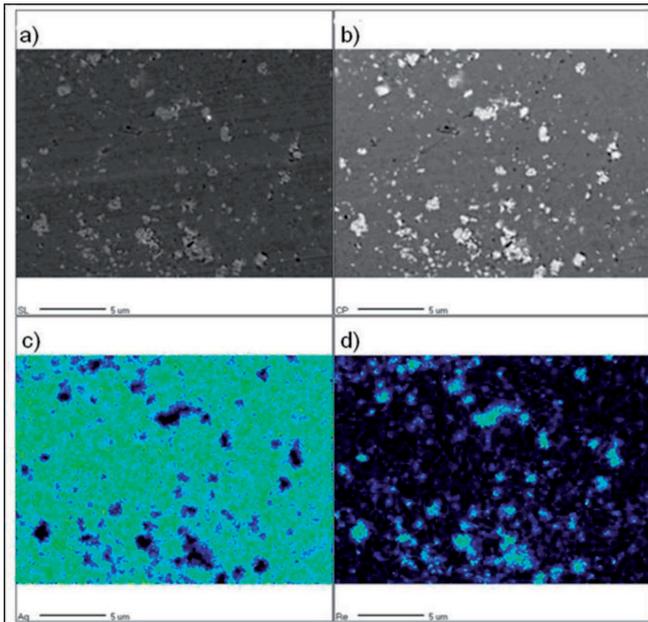


Fig. 12. SEM image of the AgRe10 powder after 150 hours of milling: (a) SEI, (b) COMPO, and distribution maps for the elements: (c) Ag, (d) Re

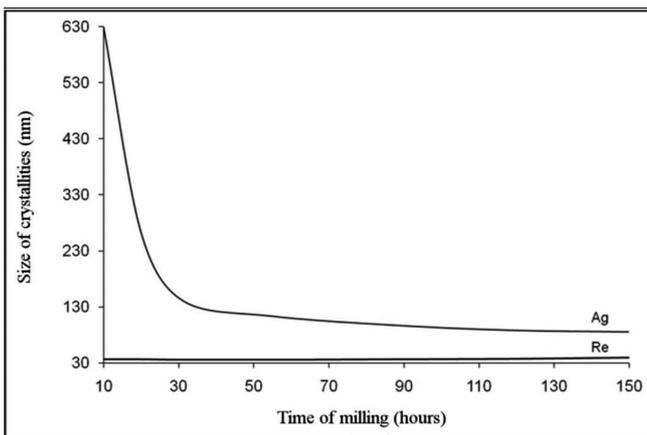


Fig. 13. Size of crystallites in a mixed powder of silver and rhenium in dependence on time of milling in high-energy ball mill

The tests showed that it was not possible to get an alloyed phase of silver with rhenium, whereas severe deformation particularly of a silver powder resulted in considerable reduction in a size of crystallites within silver grains, which is illustrated in Fig. 13. It should be emphasised that the size of these crystallites decreases exponentially with time to about 86nm after 150 hours of milling, but in case of rhenium the milling time has no clear effect on the size of Re crystallites.

## 2.2. Pressure Consolidation and Plastic Consolidation in the Processes of Extrusion by KOBO System Followed by Drawing

The flake-like shape of silver powders with embedded rhenium grains illustrated in Fig. 11 and 12 created a serious problem during pressure consolidation. The process was conducted using isostatic press in latex mulda with an internal diameter of about 18 mm, and the compacts obtained were of irregular shape and had low density. Their sintering at different temperatures for various periods of time did not result in reaching required properties such as density at the level of 6.68 g/cm<sup>3</sup> and electrical conductivity at the level of 27.78 MS/m. The compacts of similar chemical composition made from a composite material obtained by classical powder metallurgy were reportedly exhibiting higher level of these properties, i.e. 9,44 g/cm<sup>3</sup> and 38,88 MS/m, respectively [4]. Under these circumstances it was decided to intensify the process of plastic consolidation of these compacts subjecting them to extrusion on horizontal hydraulic press operating under the KOBO system. The KOBO press and schematic diagram of the extrusion process conducted by means of this press is shown in Fig. 14.

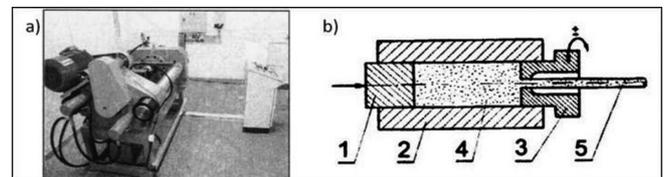


Fig. 14. The KoBo press: (a) general view; (b) principle of operation: 1 – punch, 2 – container, 3 – die rotating on both sides, 4 – starting material, 5 – output product

TABLE 4

Physical and mechanical properties of the AgRe10 compacts after consolidation by deformation and sintering followed by extrusion in a KoBo press

	Extrusion ratio $\lambda$	Density, g/cm <sup>3</sup>	Conductivity, MS/m	$R_m$ MPa	$R_{0,2}$ MPa	$A_{11,3}$ MPa	$HV_1$
Compacts after consolidation by deformation and sintering							
Mixing and pressure consolidation	–	9,44	38,88	–	–	–	–
Milling in high-energy ball mill and pressure consolidation	–	6,68	27,88	–	–	–	–
Wire after extrusion in KoBo press							
Mixing and pressure consolidation	20	10,71	52,95	192,2	133,3	14,5	64,97
Milling in high-energy ball mill and pressure consolidation	52	10,53	50,32	280,3	279,4	5,0	95,88

Chemical composition of the powders of silver, tin oxide and bismuth oxide

Material	Ag	Cu	Fe	Pb	Zn	Sn	Bi	O	Si	C	P
	% wt										
Ag	reszta	0,0038	0,0049	<0,0002	<0,0003	–	–	–	–	–	–
SnO <sub>2</sub>	–	–	–	–	–	78,0	–	22,0	0,03	0,01	–
Bi <sub>2</sub> O <sub>3</sub>	–	–	0,03	–	–	–	89,5	10,4	0,01	0,06	0,02

The compacts about 18 mm in diameter were subjected to single-hole extrusion into wire 2.50 mm in diameter. The extrusion ratio  $\lambda$  was equal to 52, i.e. it was much higher than that used during consolidation of Ag-Re10 composite with classical microstructure. Physical and mechanical properties of wires obtained by this method are presented in Table 4 together with respective comparative literature data given in [4]. Microstructure of the wires obtained was examined mainly in order to confirm its nano-structural character, and analysis of grain boundaries between silver and rhenium was made hoping to find a new alloy phase of both these metals. The results of these studies are presented in Fig. 10-13. The extruded wires were subjected to softening annealing before further drawing to the final diameter of about 1.90 mm, and to additional processing to the size enabling using them for the production of solid and bimetallic contact rivets.

### 3. Preliminary results of tests related to fabrication of the Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub> nanocomposite – mechanical synthesis of a mixture of powders of silver and oxides of tin and bismuth

The Ag-SnO<sub>2</sub>Bi<sub>2</sub>O<sub>3</sub> nanocomposite contact material has been fabricated using the powders of silver and oxides of tin and bismuth. Their main physical and chemical properties are presented below. Physical and technological properties of the powders are presented in Table 5. The microstructure examination showed that the grain size of commercially available oxides is nanometric, which demonstrate the data given in Table 6. In Fig.15-17 results of morphology examination are presented.

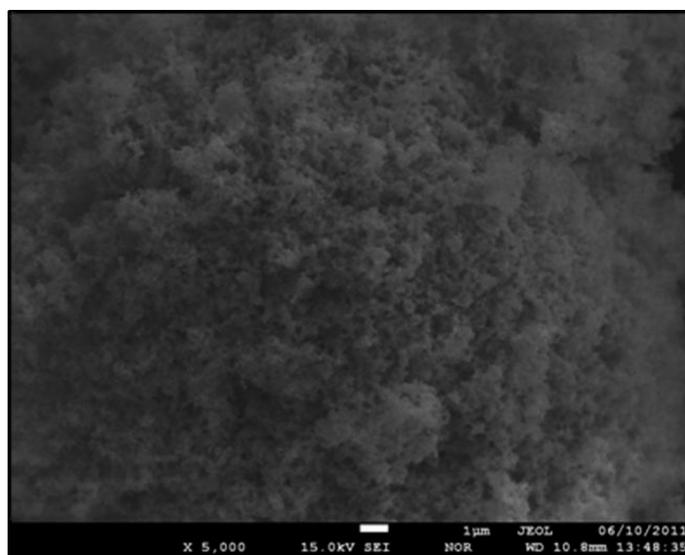
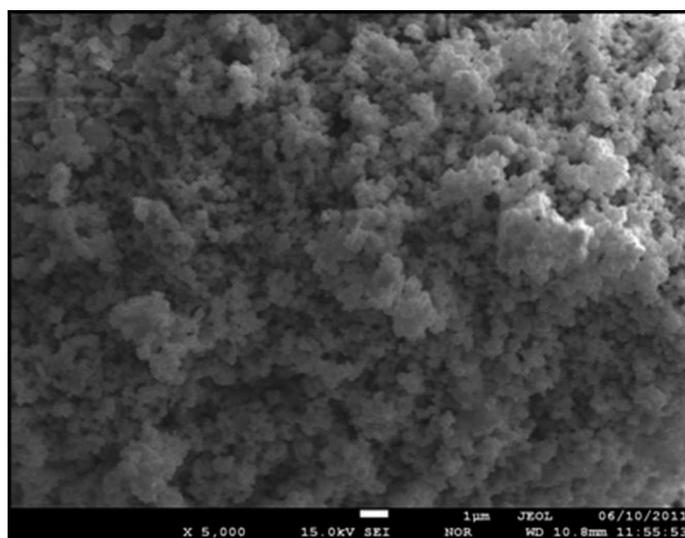
TABLE 6

Physical and technological properties of the powders used

Material	Density, g/cm <sup>3</sup>	Bulk density, g/cm <sup>3</sup>	Specific surface area, m <sup>2</sup> /g	Median grain size, nm
Ag	10,40	4,20	0,06	18740
SnO <sub>2</sub>	6,86	0,49	7,79	290
Bi <sub>2</sub> O <sub>3</sub>	8,90	1,31	3,04	310

A powder mixture was prepared as a reference material for high-energy milling; the contents of particular components being the following: Ag = 90 wt%, SnO<sub>2</sub> = 9.6 wt%, and Bi<sub>2</sub>O<sub>3</sub> = 0.4 wt%. Composition of this mixture was examined using the Electron Probe X-ray microanalyser. It was decid-

ed to conduct high-energy milling for 150 hours using the same equipment as that applied for the synthesis of Ag-Re10 nanocomposite. After 30 hours of milling powder samples were taken in order to assess an advancement in nanocomposite formation. The results obtained are presented in Fig. 18 and 19.

Fig. 15. Morphology of the SnO<sub>2</sub> powderFig. 16. Morphology of the Bi<sub>2</sub>O<sub>3</sub> powder

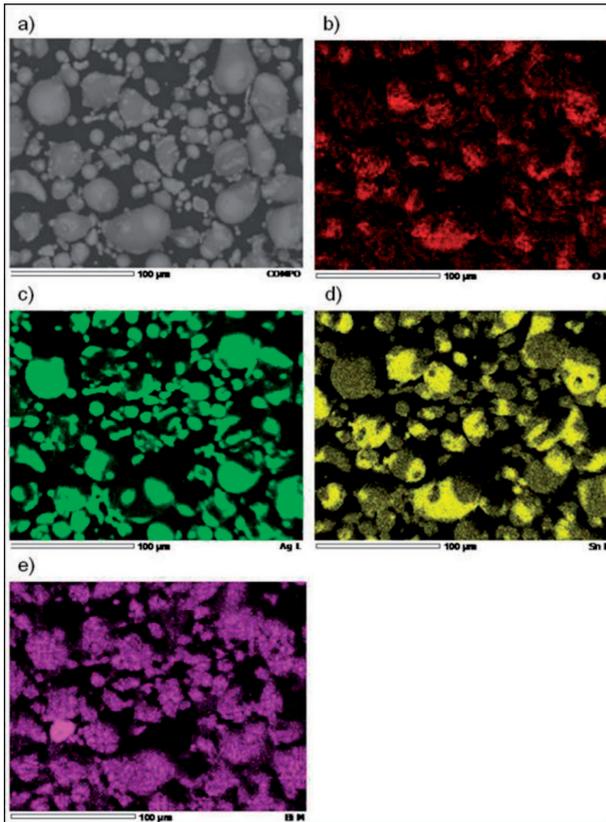


Fig. 17. SEM image of elements distribution in the  $\text{Ag-SnO}_2\text{Bi}_2\text{O}_3$  mixed powder before the milling process: a) COMPO, b) oxygen O, c) silver Ag, d) tin Sn, e) bismuth Bi

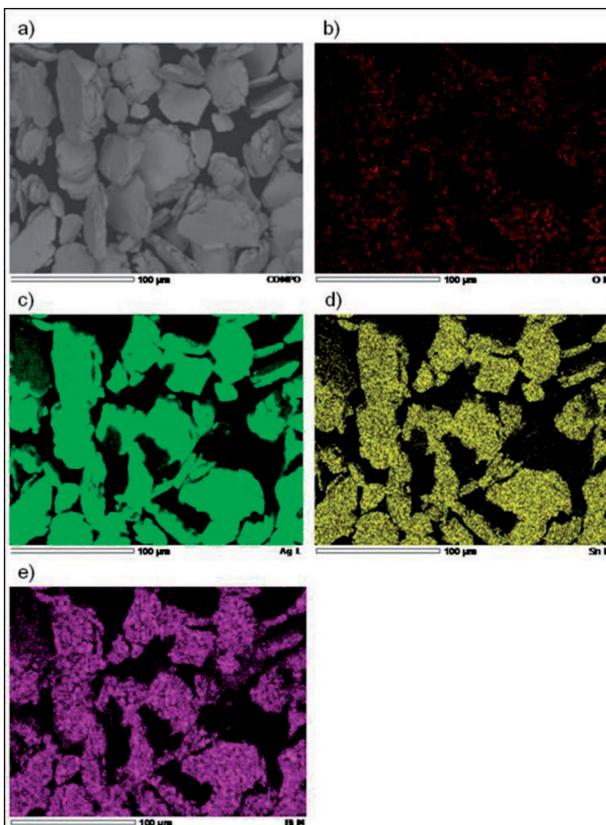


Fig. 18. SEM image of elements distribution within the  $\text{Ag-SnO}_2\text{Bi}_2\text{O}_3$  powder mixture after 30 hours of milling: a) COMPO, b) oxygen O, c) silver Ag, d) tin Sn, e) bismuth Bi

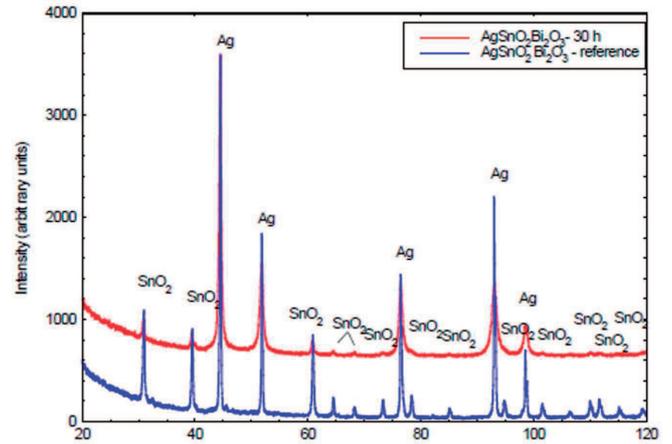


Fig. 19. Diffraction patterns obtained for the  $\text{Ag-SnO}_2\text{Bi}_2\text{O}_3$  powder mixture before milling and after 30 hours of milling in high-energy ball mill

#### 4. Analysis of results

High-energy milling of a mixture of silver powder with rhenium powder conducted for 150 hours caused very high degree of plastic deformation of a silver powder, changing its shape from spheroidal into flake-like one. The rhenium grains were embedded into the flakes, which was revealed during X-ray microanalysis (see Fig. 11-12). It was also found that microstructure of silver powder flakes was very refined, and the size of silver crystallites decreased to 85nm after 150 hours of milling. Similar examination was made for rhenium powder showing that the size of crystallites before and after high-energy milling was at the similar level of 42-32 nm. During the tests with pressure consolidation of the flake-shaped powder conducted by means of isostatic press serious technological problems were encountered. The compacts had irregular shape, considerably differing from cylindrical shape, and cracks could be found on them. Their density was at the level of  $6.68 \text{ g/cm}^3$ , i.e. considerably lower than density of the compacts fabricated from powders of similar chemical composition subjected to mixing by conventional method, which is equal to  $9.44 \text{ g/cm}^3$ .

After sintering, the compacts fabricated under this work had electrical conductivity at the level of 27.88 MS/m, which was by about 11.00 MS/m less than conductivity of similar compacts obtained under the work [3]. However, it was expected that after plastic consolidation of these compacts conducted by extrusion in horizontal hydraulic press operating under the KOBO system and at higher extrusion ratio of  $\lambda = 52$ , the physical and mechanical properties of the produced wire will be significantly improved (it should be noted that under the work [4] the compacts were fabricated at the extrusion ratio of 20). This expectation was confirmed in case of wires obtained from nanocomposite Ag-Re, since electrical conductivity and density of these wires were at the level of 50.32 MS/m and  $10.53 \text{ g/cm}^3$ , respectively, i.e. at the levels comparable with those for the wire 4 mm in diameter fabricated from powders mixed by conventional method. Other mechanical parameters such as tensile strength, yield strength and hardness, presented in Table 4, were the following: wire 2.5 mm in diameter –  $R_m = 280.3 \text{ MPa}$ ,  $R_{0.2} = 279.4 \text{ MPa}$ ,  $A_{11.3} = 5.0\%$

$HV_1 = 95,88$ ; wire 4 mm in diameter –  $R_m = 243.3$  MPa,  $R_{0.2} = 224.3$  MPa,  $A_{100} = 6.8\%$  and  $HV_1 = 64.97$ .

Comparison of microstructure of both types of wires was also made showing that the 2.5 mm wire was nanostructured material, so it should be expected that the contact rivets made from this material will exhibit considerably enhanced electrical parameters compared to those characterising similar products fabricated from mechanical mixture of the powders of silver and rhenium having classical microstructure. Similarly, the results obtained for the  $AgSnO_2Bi_2O_3$  powders subjected to high-energy milling were positive, since this process resulted in high degree of deformation in silver powders and in the formation of nanostructure, which has been shown in Fig. 18-19.

### 5. Conclusions

At the present stage of research related to the development of fabrication technology of nanocomposite contact materials Ag-Re10 and  $AgSnO_2Bi_2O_3$  the following conclusions can be formulated:

1) High-energy milling of mixed powders of silver and rhenium leads to high plastic deformation particularly of silver powder grains, which results in considerable refinement of its structure and makes it possible to obtain the size of crystallites at the level of 86 nm.

2) The mechanical synthesis of the powders of silver and rhenium conducted under pre-designed technological conditions (high-energy ball mill, balls diameter of 10 mm, a ratio of ball mass to the mass of powder mixed equal to 10, and rotary speed of a drum reaching about 55 rev/min) did not result in the formation of the alloyed phase of both metals.

3) As a result of severe plastic deformation of silver powder during high-energy milling the shape of this powder changes from spheroidal to a flake-like, with embedded rhenium grains; a size of crystallites being at the level of 30 nm.

4) Plastic consolidation of compacts fabricated from silver powder flakes with the embedded rhenium grains, taking place during extrusion conducted in hydraulic press operating under the KOBO system, appeared to be very effective operation of plastic working ensuring relatively high density of the fabricated wires and their high electrical conductivity.

5) High-energy milling of the powders of silver, tin oxide and bismuth oxide, conducted in similar conditions as in the case of Ag-Re10 material, resulted in severe plastic deformation of a silver powder changing its shape from spheroidal into flake-like and embedding the oxides  $SnO_2$  and  $Bi_2O_3$  into those flakes.

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