A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
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SILVER – BASED INFILTRATED COMPOSITES

INFILTROWANE KOMPOZYTY NA OSNOWIE SREBRA

Attempts have been made to describe the influence of production process parameters on the microstructure and properties of W – Ag and Mo – Ag composites. W-30%Ag, W-40%Ag, Mo-30% Ag and Mo-40%Ag composites were produced by tumbling W5%Ag and Mo-5%Ag mixtures for 30 minutes in the Turbula T2F mixer. The powders were subsequently cold pressed in a rigid die on a single action press. The pressure was adjusted individually to assure 25% and 35% green porosity. Prior to infiltration the compacts were sintered in hydrogen at 1100°C for one hour. The porous skeletons were finally contact infiltrated with Ag-1%Ni alloy at 1100 and 1200°C for one hour in hydrogen. The main objective of this work was to compare properties and microstructure of the as-infiltrated composites.
Keywords: composites, tungsten, molybdenum, silver, sintering, infiltration

Srebro jest pierwiastkiem krystalizującym w sieci A1; jest metalem bardzo plastycznym na zimno, posiada niską twardość ~26 HB, stosunkowo wysoką gęstość 10,5 g/cm^3 i niską temperature topnienia 960°C, jest odporne na działanie czynników atmosferycznych, jest najlepszym przewodnikiem cieplnym i elektrycznym. Wytrzymałość na rozciąganie srebra jest mała i wynosi około 160 MPa. Proszek srebra wytwarza się metodą elektrolizy lub rozpylania. Czyste srebro do celów konstrukcyjnych nie jest stosowane, ze względu na niskie własności wytrzymałościowe. W procesie wytwarzania metodą metalurgii proszków styków elektrycznych należy dążyć do uzyskania materiału o drobnoziarnistej strukturze i równomiernym rozmieszczeniu składników, z których najczęściej jeden jest odpowiedzialny za wysokie przewodnictwo, natomiast drugi źle przewodzący prąd zwiększa odporność mechaniczną, odporność na zgrzewanie i elektroerozję. W zależności od udziałów objętościowych składników dąży się albo do uzyskania materiału, którego strukturę cechuje równomierne rozmieszczenie cząstek jednego składnika w osnowie składnika podstawowego, albo w przypadku zbliżonych zawartości składników – materiału złożonego z dwu przenikających się składników.

Celem niniejszego opracowania jest przedstawienie wyników badań dotyczących formowania metodą infiltracji oraz spiekania w stanie stałym kompozytów wolfram-srebro oraz molibden-srebro. Do formowania kompozytów stosowano elektrolityczny proszek srebra, redukowany proszek wolframu oraz redukowany proszek molibdenu. Materiał badawczy stanowiły kształtki wolframu z dodatkiem 30% i 40% srebra oraz molibdenu z dodatkiem 30% i 40% srebra. Porowate kształtki przeznaczone do infiltracji prasowano na stałą objętość oraz wstępnie spiekano w atmosferze wodoru w temperaturze 1100°C przez 60 minut. Następnie porowate kształtki infiltrowano srebrem z dodatkiem 1% niklu, metodą nakładkową w atmosferze wodoru w temperaturze 1100 i 1200°C przez 60 minut. Kompozyty poddano badaniom stopnia wypełnienia kapilar, gęstości względnej, twardości, wytrzymałości na zginanie, własności tribologicznych oraz przewodności elektrycznej i cieplnej w odniesieniu do czystego srebra. W pracy przedstawiono także struktury kompozytów otrzymanych w wyniki infiltracji.

W wyniku infiltracji można formować kompozyty wolfram-srebro oraz molibden-srebro odznaczające się względnie małą porowatością (mniejszą od 7%), oraz dobrymi własnościami wytrzymałościowymi. Z punktu widzenia ekonomicznego oraz w wyniku analizy własności otrzymanych kompozytów można stwierdzić, że bardziej korzystne wydaje się zastosowanie temperatury infiltracji 1100°C.

1. Introduction

Electrical contact materials are used in a variety of applications, such as electrical switches, contactors, circuit breakers, voltage regulators, arcing tips, switch gears and relays. Materials used for electrical contacts must have a combination of high electrical conductivity, resistance to erosion, welding and mechanical wear. When using contact materials in heavy duty applications, it is very important to control the erosion which takes place as a result of electrical arcing, and the danger of welding due to contact heating during current flow and arc

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formation. When breaking the circuit an arc forms between the contacts. The cross-section of the conducting area initially decreases and current density, contact voltage and, consequently, temperature increases causing the contact material to melt locally, and the current passes only across a small molten metal bridge. Thus droplets of molten contact material solidify as they cool forming contact debris. When closing the circuit the moving contact may bounce against the stationary one thus causing the arc to reappear many times.

The desired combination of properties can be realized only by utilizing refractory metals, such as tungsten or molybdenum. The W-Ag and Mo-Ag electrical contact materials utilised in heavy-duty applications are produced exclusively by means of powder metallurgy (PM) [1÷6]. Composites containing 30-80 wt-% silver resist arc erosion and possess good welding resistance and current carrying capacity. The properties of PM electrical contacts depend on both their composition and manufacturing process. The particle size and distribution of the refractory phase, homogeneity of microstructure and amount of porosity combine to affect the electrical, mechanical, and thermo-physical properties of the composite material [1,2,6].

In principle, W-Ag and Mo-Ag composite materials can be produced by two techniques [1,4,5,6], which consist in:

- mixing elemental powders, cold pressing and solid state sintering. At silver contents exceeding 30% the as-sintered parts can be subjected to final processing by rolling or re-pressing
- infiltrating tungsten, or molybdenum, porous skeletons with liquid silver.

Infiltration is a process that has been broadly used for many years. It is defined as a process of filling the pores of a sintered or unsintered compact with a metal or alloy having a lower melting temperature. While it is possible with the first method to produce materials with any ratio of high-melting point and low-melting point components, the infiltration process is suitable only for materials with tungsten or molybdenum content higher than 70%. In practice this limitation has little significance, because the materials used most commonly are highly burn resistant and have a higher W/Mo content anyway. For this reason and because of their excellent application properties, infiltrated contacts predominate.

For many years, tungsten has been used as the contact material for automotive distributor ignition system, aviation and diesel starting equipment, car radios, spark gap electrodes and in a majority of automotive voltage regulators. Molybdenum is used when electrical loads do not justify the use of tungsten. Molybdenum has lower density than tungsten which is advantageous in situations where the mass of moving parts of the contact must be minimised. Molybdenum-base contacts are used for breaking lower currents and in high voltage electronics, whereas tungsten is preferred in car electronics, as contact-breaker points, for horns, etc [1÷8].

2. Experimental procedure

The starting powders are characterised in Fig. 1 and in Table 1.

TABLE 1

Selected properties of the experimental powders

Powder	Particle size, μm	Average particle size, μm	Tap density, g/cm^3	Flow time, s/50g	Compressibility at 600 MPa, g/cm^3	Theoretical density, g/cm^3
W	0-10	3.1	2.82	-	12.54	19.3
Mo	0-10	3.1	1.64	-	7.21	10.2
Ag	0-63	32.8	1.53	60	9.76	10.5
Ni	0-40	20	2.32	-	6.54	8.9

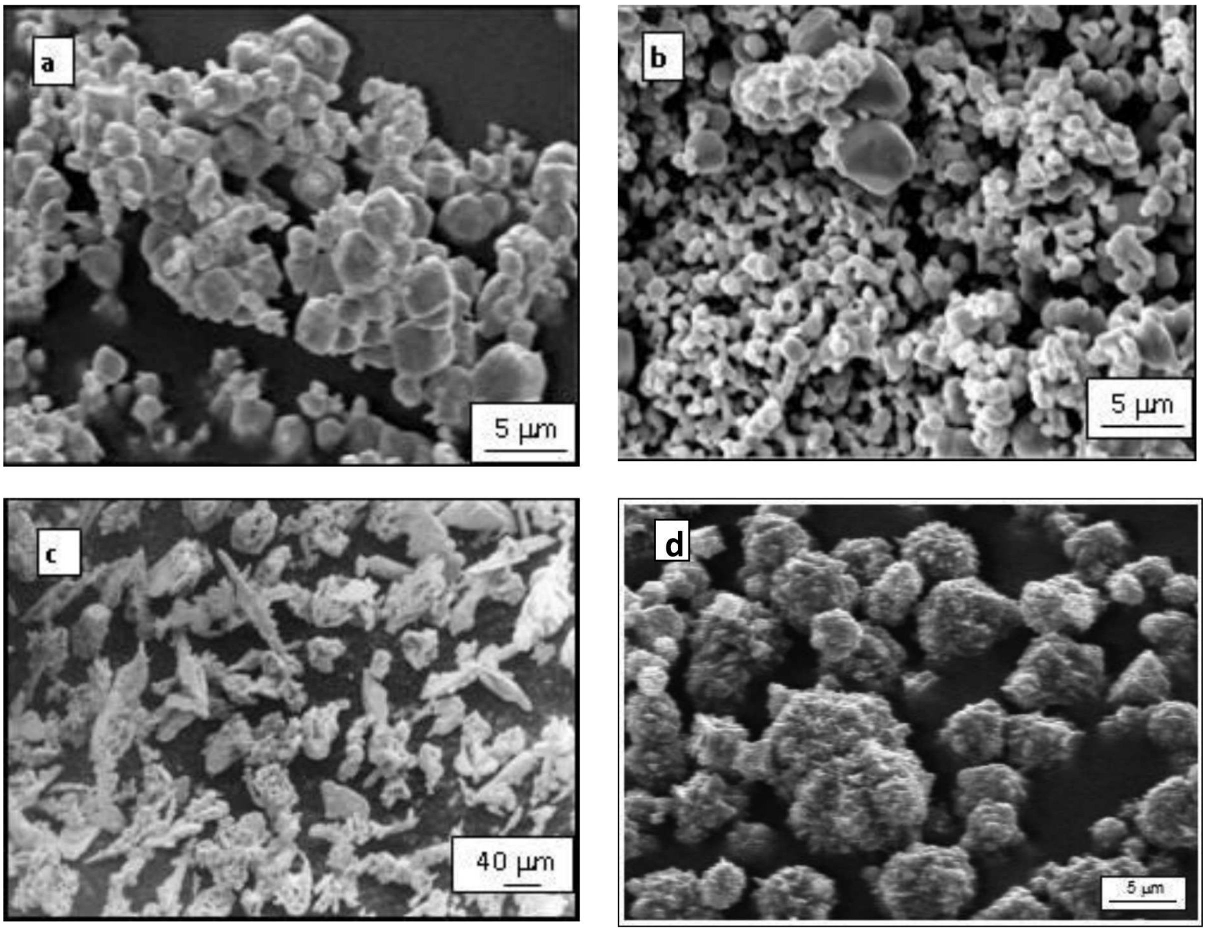

Fig. 1. SEM micrographs of: a) tungsten, b) molybdenum, c) silver and d) nickel powders

W+5wt-%Ag and Mo-5wt-%Ag mixtures were prepared by tumbling the powders for 30 minutes. They were subsequently cold pressed in a rigid die on a single action press. The pressure was adjusted individually to assure around 25% green porosity. Prior to infiltration the compacts were sintered for one hour at 1100°C in hydrogen. The porous skeletons were then contact infiltrated with Ag-1wt-%Ni alloy by holding either at 1100 or 1200°C.

The infiltrated specimens were subsequently tested for density, Brinell hardness, bending strength and resistance to wear, and subjected to microstructural examinations by means of both light microscopy (LM) and scanning electron microscopy (SEM). The wear tests were carried out using the block-on-ring tester (Fig. 2).

During the test a rectangular 20×4×4 mm wear sample (1) was mounted in a sample holder (4) equipped with a hemispherical insert (3) ensuring proper contact between the test sample and a steel ring (2), heat treated to 55 HRC, which was rotated at a constant speed of 136 rpm. The wear surface of the sample was perpendicular to the loading direction. Double lever system was used to force the sample towards the ring at 56 N ± 1%.

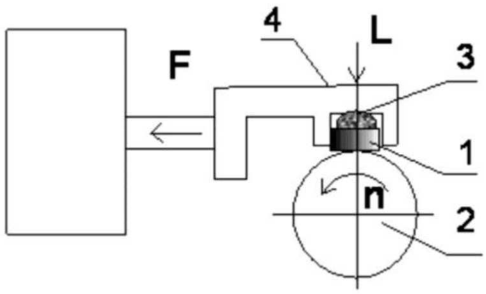

Fig. 2. Schematic view of a block-on-ring tester

The loss of sample mass was measured after a sliding distance of 100 m. The friction force *F* was also measured and used to calculate the friction coefficient.

The electrical conductivity of contact materials was carried out on electrical conductivity measurement device (Fig. 3).

The conductivity of the annealed copper is 5.8×10^7 S/m and copper is defined to be 100% IACS at 20°C. All other conductivities are compared to annealed copper and expressed in relative values.

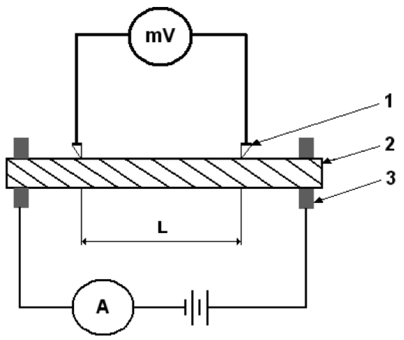

Fig. 3. Schematic view of the electrical conductivity measurement device: 1 – electric resistance tester, 2 – sample, 3 – grip spring

3. Results and discussion

The combined effects of the powder mix composition and its processing route on relative densities of the composites are shown in Fig. 4.

The other properties of contact infiltrated composites are given in Figs 5÷7.

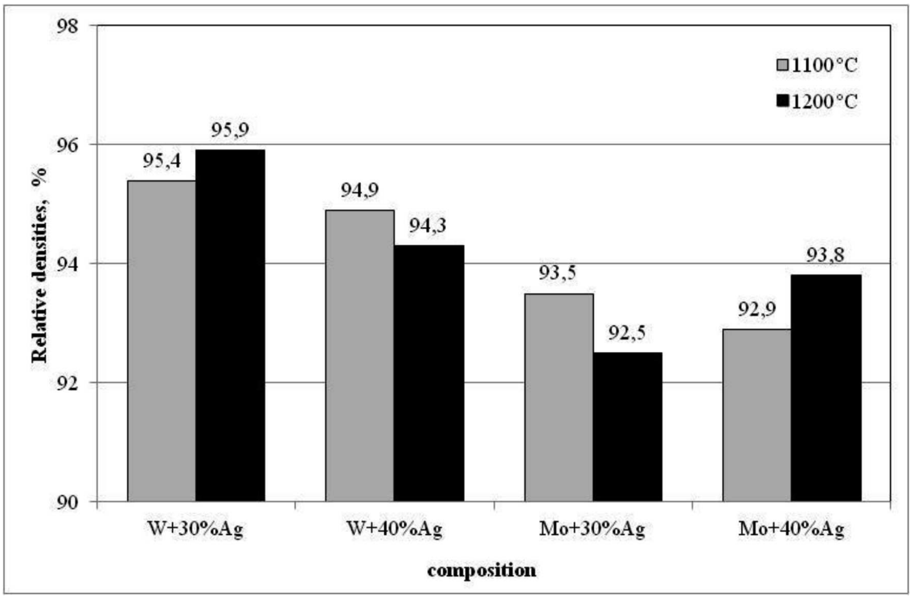

Fig. 4. Relative densities of composites as a function of composition and temperature of infiltration

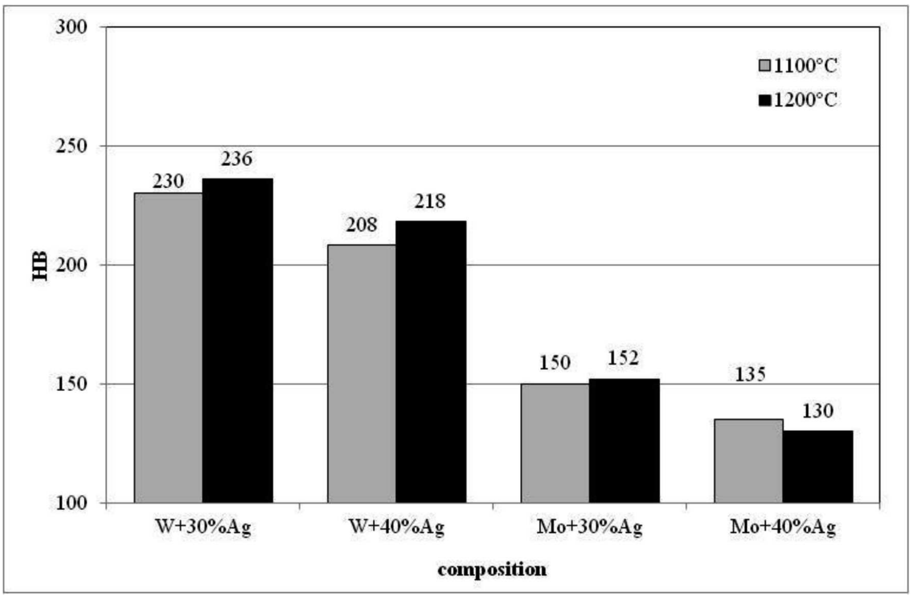

Fig. 5. Brinell Hardness of composites as a function of composition and temperature of infiltration

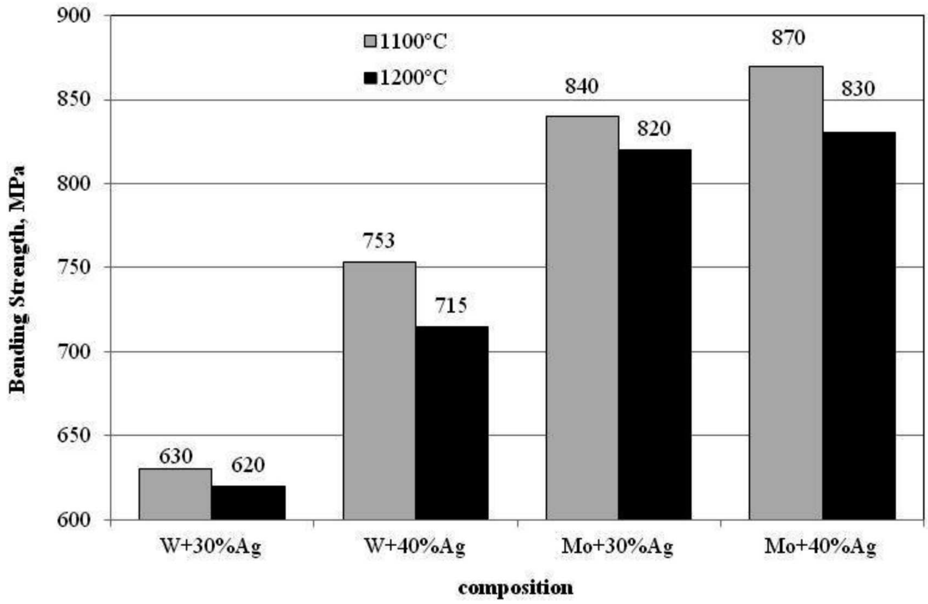

Fig. 6. Bending Strength of **c**omposites as a function of composition and temperature of infiltration

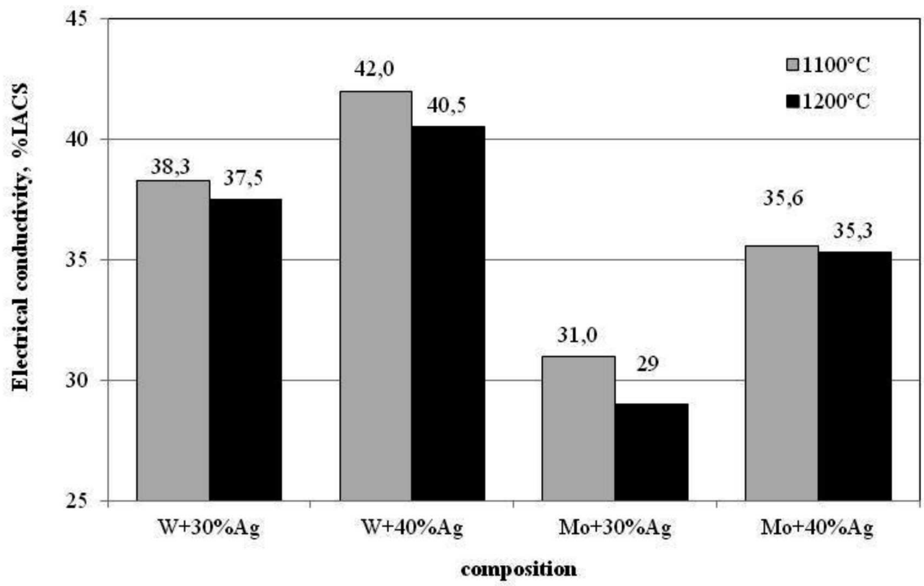

Fig. 7. Electrical conductivity % IACS of **c**omposites as a function of composition and temperature of infiltration

As seen in Figure 4 the molten silver was drawn into the interconnected pores of the skeletons, through a capillary action, and filled virtually the entire pore volume to yield final densities exceeding 92.5÷96% of the theoretical value.

It can be seen that hardness of Ag-W and Ag-Mo electrical contact materials (Fig. 5) increased with increasing W/Mo content, but its electrical conductivity (Fig. 7) and bending strength (Fig. 6) decreased. Silver imparts conductivity while tungsten and molybdenum strengthen the composites. The electrical conductivity of both Ag–W and Ag–Mo composites strongly depend on the volume fraction of silver because there is practically no mutual solubility of the components. The hardness of W+30%Ag composite (~230HB) is higher than that of Mo+30%Ag (150HB), and its electrical conductivity is higher as well. Tungsten does not oxidize as readily as molybdenum. Thus tungsten-based materials offer lower surface resistance compared to their molybdenum-based counterparts.

It can be seen in Figs 5 and 7 that infiltration at 1200°C results in lower electrical conductivity and bending strength. The values of electrical conductivity and hardness obtained from the present study are consistent with the values given in the literature [3, 9, 10].

The wear test results of composites infiltrated at 1100°C are given in Figures 8 and 9.

It has been found that the tribological behavior of the investigated composites depend on the chemical composition of the material, and the wear resistance was higher for the W-Ag materials. Interestingly, the lowest loss of mass was observed in the W+40%Ag composites. This may be attributed to good sliding properties.

Characteristic surface topographies after the wear test are presented in Fig. 10.

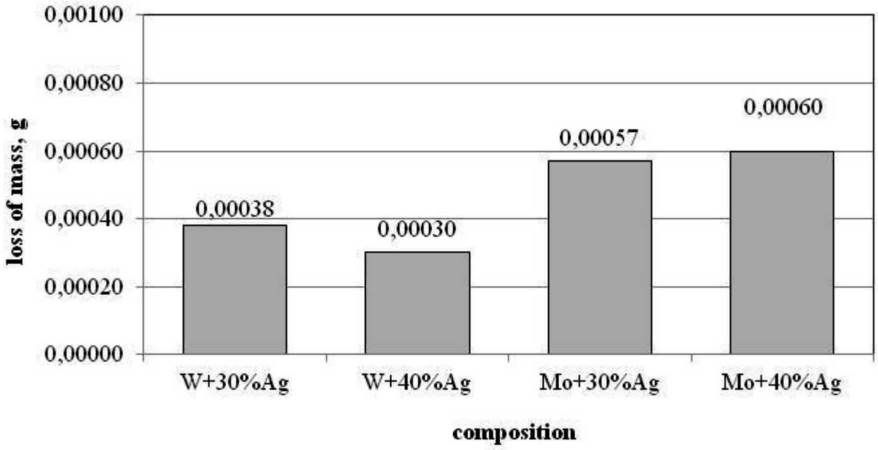

Fig. 8. Loss of mass of as infiltrated composites

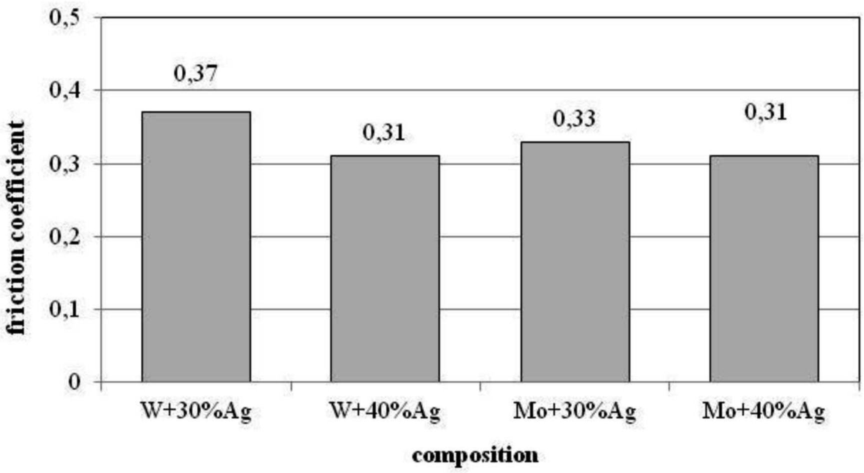

Fig. 9. Friction coefficient of as infiltrated composites

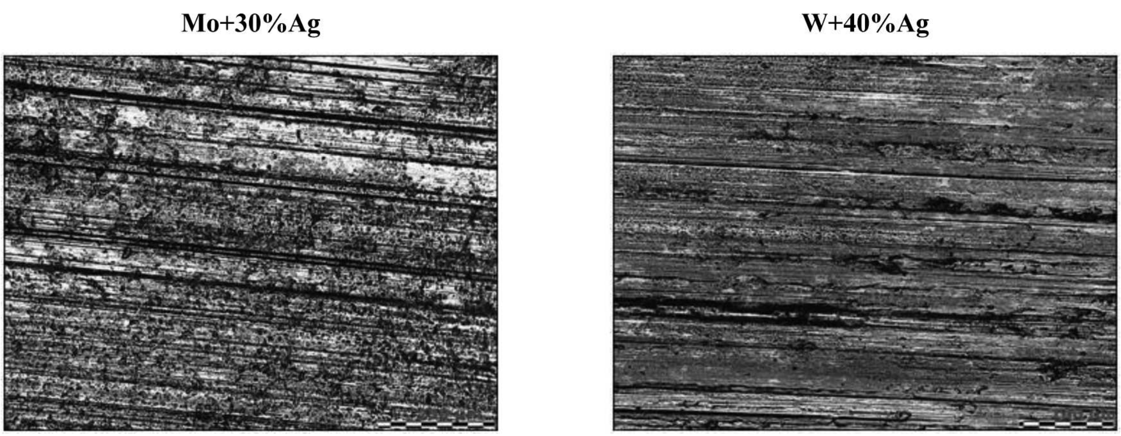

Fig. 10. The surface topographies of selected composites after the wear test

The surface topographies of Mo+30%Ag and W+40%Ag specimens give evidence of massive micro-ploughing by Mo and W particles pulled out of the matrix to act as abrasives. This seems to contribute to the increase in the coefficient of friction. Fig. 10 also shows smearing of silver over the surface of the as-infiltrated composites which implies a marked contribution of adhesive wear.

The microstructures of as-infiltrated W-Ag and Mo-Ag composites in Figs 11-14 illustrate uniform distribution of tungsten/molybdenum (dark) and silver (gray), and small residual porosity, although it seems that molybdenum powder particles show higher tendency to form bigger agglomerates than tungsten.

T_i = 1100°C T_i = 1200°C

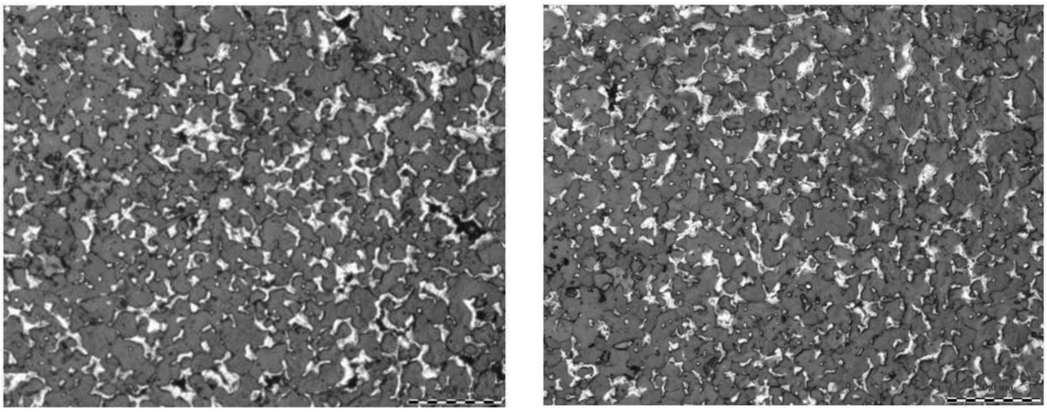

Fig. 11. Microstructures of as-infiltrated W+30%Ag composites

T_i = 1100°C T_i = 1200°C

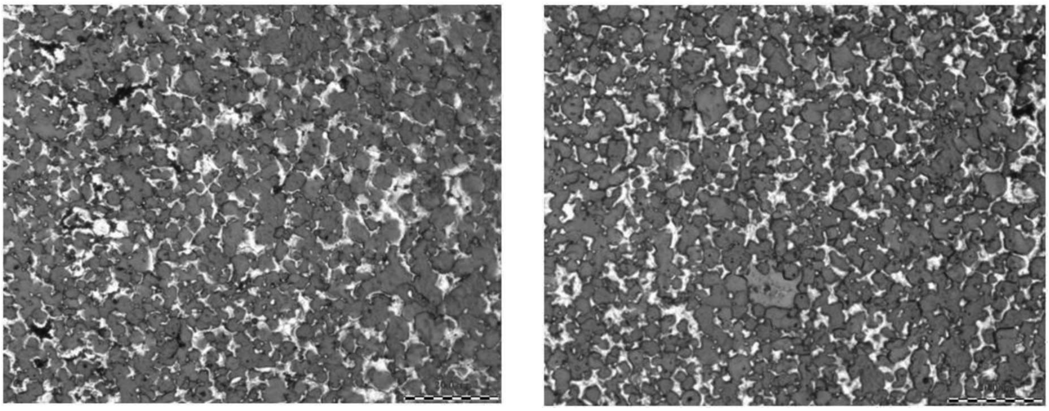

Fig. 12. Microstructures of as-infiltrated W+40%Ag composites

T_i = 1100°C T_i = 1200°C

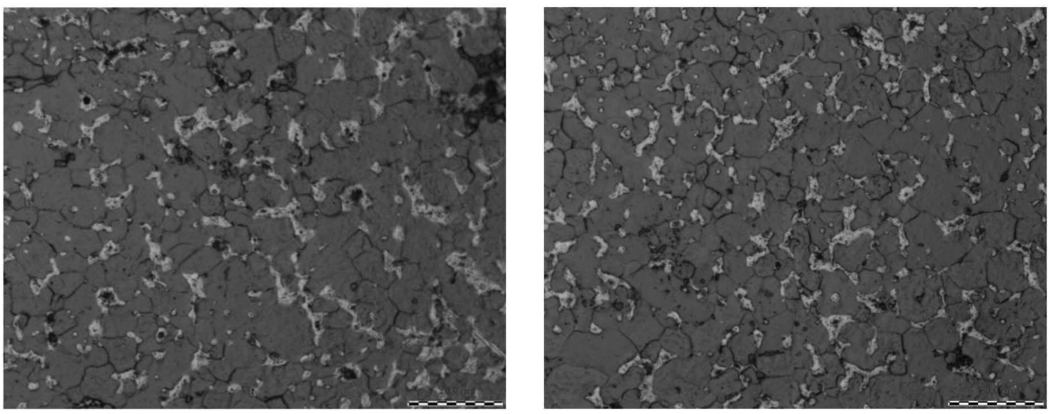

Fig. 13. Microstructures of as-infiltrated Mo+30%Ag composites

$T_i = 1100°C$ $T_i = 1200°C$

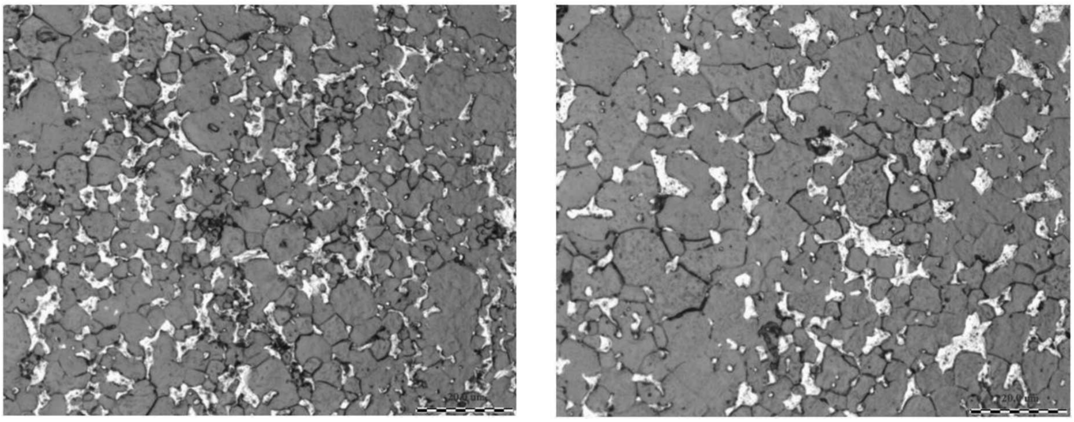

Fig. 14. Microstructures of as-infiltrated Mo+40%Ag composites

4. Conclusions

1. Infiltration of porous W/Mo skeletons with liquid silver at between 1100 and 1200°C has proved to be a suitable technique whereby fully dense electrical contact materials are produced at low cost.
2. The electrical conductivity of W-Ag and Mo-Ag composites increase with silver content.
3. The hardness of W-Ag and Mo-Ag composites increase with the amount of tungsten and molybdenum.
4. All contact materials studied in this work show relatively homogenous distribution of tungsten and molybdenum.

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