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The Influence of Tuff Particles on the Properties of the Sintered Copper Matrix Composite for Application in Resistance Welding Electrodes

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Featured Application: Welding technology.

Abstract: This paper presents modern copper-matrix composite materials in which volcanic tuff particles are used as a reinforcing phase. The aim of the research was to determine the optimal shares of volcanic tuff additive based on such criteria as softening temperature, relative density, electrical conductivity, and hardness. The properties of the produced and tested composites allowed us to determine the usefulness of this type of material for resistance welding electrodes. To confirm the assumptions made, preliminary investigations of the durability and behavior of electrodes made of the tested material during the processes of welding non-alloy steel sheets were carried out. As a result of the research, it was discovered that the addition of 5% tuff produces the best results in this type of composite. It was found that for the sample with a 5% share of tuff, a high softening point above 600 °C was obtained, high hardness after densification at the level of 62 HRB, and high relative density of approximately 95% and very good conductivity at the level of approximately 45 MS/m. The conducted tests did not show any electrode wear different from the commonly used alloys for resistance welding.

Keywords: metal matrix composites (MMC); particle-reinforced composites; volcanic tuff; softening temperature; welding electrodes

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1. Introduction

The very rapid development of composite materials has been observed over the last few decades which often offers unexpected possibilities for modeling the properties of products. A wide range of composites are metal matrix composites strengthened with reinforcement in the form of particles, fibers, or flakes [1,2]. Powder metallurgy technology, as one of the few techniques, enables the formation of metal composites with expected properties [2,3]. The application of this technology allows, for example, the elimination of problems occurring in the casting technique related to poor wettability of ceramic particles by liquid metal or the heterogeneous distribution of the reinforcement in the matrix [4–6]. An interesting group of metal composites is the copper matrix composites. Copper is the main candidate for such applications because it has the highest thermal conductivity among the construction materials [7,8]. Materials made of copper-based alloys and composites are irreplaceable in metal joining technology [9,10]. Materials bonding is a field of material engineering in which an important role is played by resistance welding processes that are used to join elements in the automotive, household appliance, or aerospace industries. Electrodes are used in welding processes to exert pressure and supply current. They are required to have a high electrical and thermal conductivity, high softening temperature, high hardness at welding temperature, and low manufacturing costs [11,12]. High-durability electrodes are a highly desirable product of great importance and application in the industry [13,14]. The most durable electrodes available on the market are copper electrodes strengthened with dispersion aluminum oxide (Al_2O_3), manufactured using powder metallurgy technology. Copper compounds reinforced with ceramic particles such as SiC, Al_2O_3, B_4C, and TiC can also be produced by friction stir processing [14,15].

Research results indicate that copper composites with a high content of TiC reinforcing particles have good mechanical properties, such as hardness and wear resistance, while density and thermal conductivity drastically decrease. Therefore, composites with a TiC content below 5% by volume are used for applications such as spot-welding electrodes and sliding contacts [16,17]. An increase in mechanical properties and abrasion resistance was also confirmed in copper composites with the addition of carbon nanotubes [18]. Moreover, an investigation of copper matrix composites reinforced with ceramics in the form of TiB whiskers and TiB_2 particles showed that the composite containing 0.9% (w/w) TiB whiskers and 1.0% (w/w) TiB_2 has higher strength and higher electrical conductivity compared to a composite containing only 2.6% (w/w) TiB_2 particles [19].

It is extremely important that introduced ceramic particles are not dissolved in the matrix because it may significantly reduce the electrical conductivity of the composite. In Cu-SiC composites, such a negative effect can occur due to the silicon dissolution process in copper [20]. In order to avoid phase changes at the metal-ceramic boundary, it is possible to cover the ceramic particles, e.g., with tungsten and nickel coatings [21,22].

Tuff is a type of hard rock with high porosity. It consists of pyroclastic material (principally sand and volcanic ash) most often with an admixture of other crumb materials which they are cemented with, for example, a silica or clay binder [23–25]. Tuff also includes clay minerals and zeolites; in their structure, there are empty tubular spaces with a ground clearance of several Å [23–25].

Based on our previous studies, it was found that the material that can be used to strengthen copper is volcanic tuff from Filipowice, Poland. It consists of porous aluminosilicates with a developed surface, containing a series of hard and refractory oxides [23]. This tuff has several specific advantages such as the low price; availability; porosity, due to which it is possible to obtain a good connection with the matrix; low specific gravity, which contributes to the reduction in the mass of composites; and the high thermal properties, which allows composites to be used even at high temperatures above the melting point of copper [23–25]. Tuff has many application possibilities, including as a base material for the production of building materials (due to their lightness, insulation properties, and resistance to changing weather conditions) and a raw material for the production of ceramic materials, dark packaging glass, potassium-phosphorus fertilizers, and as peeling in the cosmetic industry, as well as an addition to paints and varnishes to improve their properties [23–27]. It can be used as a sorbent, as well as an additive to metals, which increases their anti-corrosion properties, adhesion and resistance to impact, and resistance to abrasion [28,29]. This material can also be used as a base component for the production of geopolymers (environmentally friendly construction materials, which currently find many different applications, including replacing cement in the construction industry, that are characterized by a short setting time and high properties strength and high fire resistance) [30,31].

This paper presents modern copper-matrix composite materials in which volcanic tuff particles are used as a reinforcing phase. The aim of the study was to determine, based on several criteria, the optimal shares of volcanic tuff additive, including softening temperature, relative density, electrical conductivity, and hardness. The properties of the produced and tested composites allowed us to determine the usefulness of this type of material for resistance welding electrodes. The tuff reinforcement was not previously applied in the copper matrix for this kind of product. To confirm the assumptions made, investigations of the durability and behavior of electrodes made of the tested material during the processes of welding non-alloy steel sheets were carried out.

2. Materials and Methods

2.1. Materials

The tuff used in the investigation came in the form of rock extracted from the deposits near Filipowice (region: Krzeszowice, Lesser Poland, Poland). It was crushed and then ground in an ultra-centrifugal laboratory mill RETSCH ZM1 (Retsch GmbH, Haan, Germany) to create grain sizes of less than 40 µm. It was then roasted at 850 °C for 4 h to remove moisture and organic substances that could be emitted during sintering at 900 °C and thus contaminate the process. The experimental tuff contains sanidine as the main ingredient and small amounts of minerals such as kaolinite, biotite, illite, and quartz. The existing biotite grains are up to 8 mm in size while the other components have very different sizes from a few mm to 5 cm [23–25]. The tuff is a material with high porosity of about 23% and a density of 2.63 g/cm^3 [32]. The oxide composition of the tuff used is presented in Table 1.

Table 1. Oxide composition of the used tuff [32].

SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	TiO_2	K_2O	Na_2O
56.04%	5.38%	16.73%	5.39%	0.60%	0.85%	9.16%	0.39%

Porosymmetric analyses with the BET method showed that the value of the specific surface area of the tuff in its natural form is approximately 9708 m^2/g [32]. These studies have shown that thermal processing of the tuff at 850 °C reduces the specific surface area of the tuff particles, reduces the volume of pores, and increases the average diameter of the pores, indicating the start of the sintering process [32].

To identify the components present in the tuff, and in particular, the type of carbonate binder that decays at temperatures close to 800 °C, XRD tests were carried out on a representative sample of the tuff in its natural state. The image of the registered diffractogram and the type of identified phases are presented in Figure 1 [33].

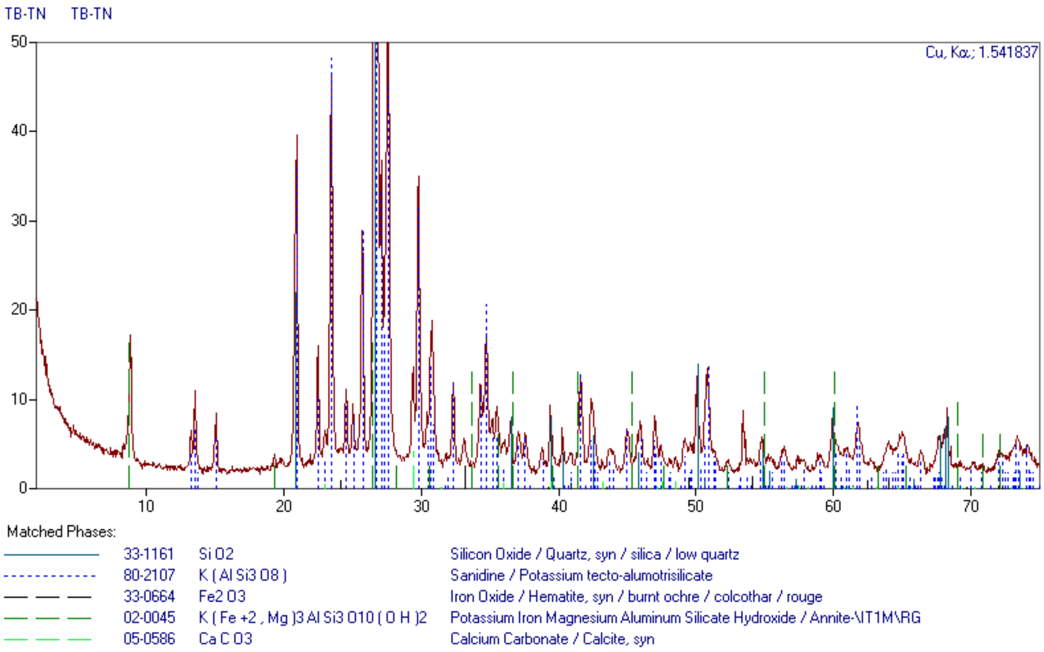
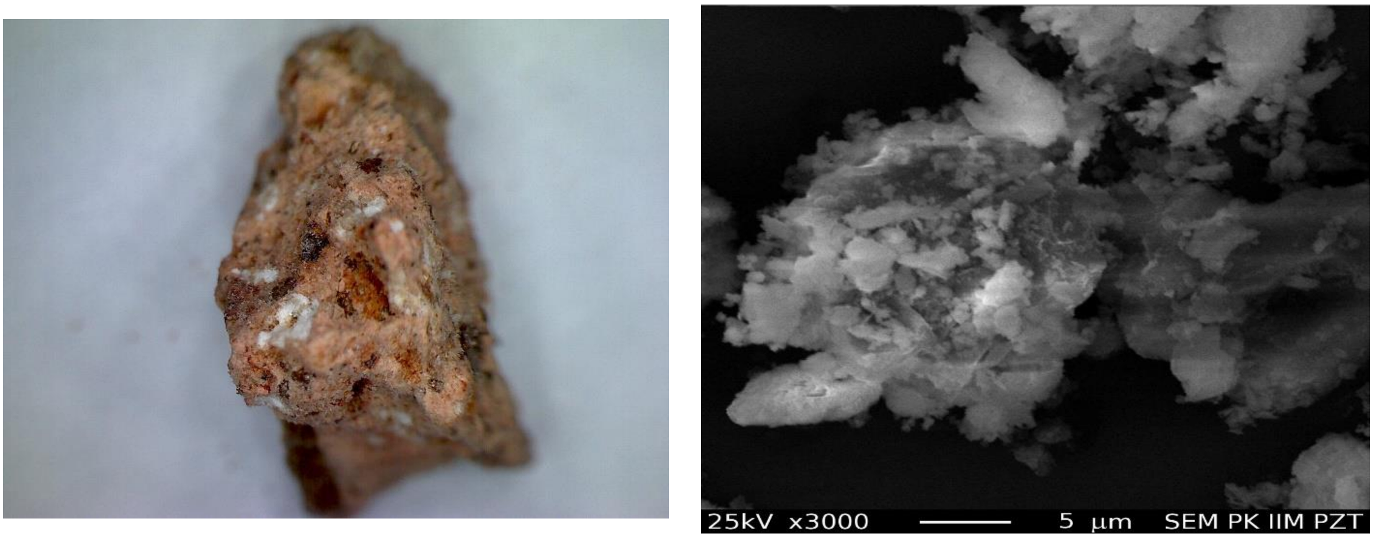


Figure 1. Results of the XRD analysis of the tuff under delivery condition: identified phases [33].

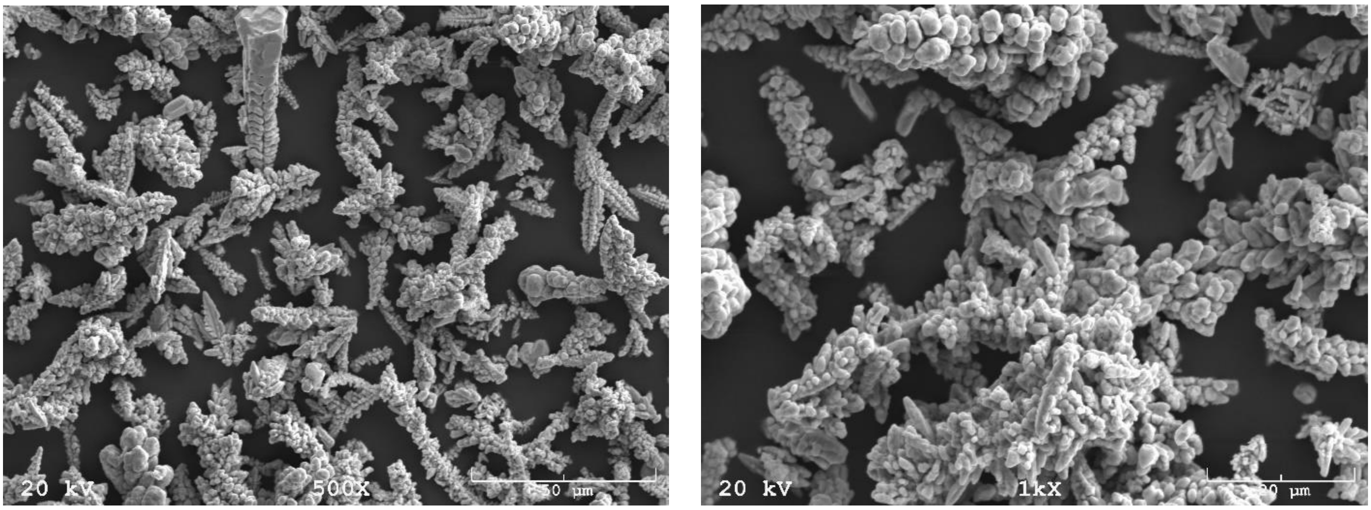
XRD research confirms that the SiO_2 and $KAlSi_3O_8$ phases previously defined in the literature, the appearance of calcium carbonate $CaCO_3$ which is a carbonate binder, was identified in the tuff.

The morphology of the tuff was analyzed on a JEOL JSM-820 (Tokio, Japan) scanning electron microscope with IXRF (IXR Inc., Austin, TX, USA). Before the test, small amounts of the materials were dried to constant weight and placed on a carbon bed. Next, the materials were sprayed with a thin layer of gold using a JEOL JEE-4X sputtering machine. The tuff was observed at high magnification—Figures 2–4.



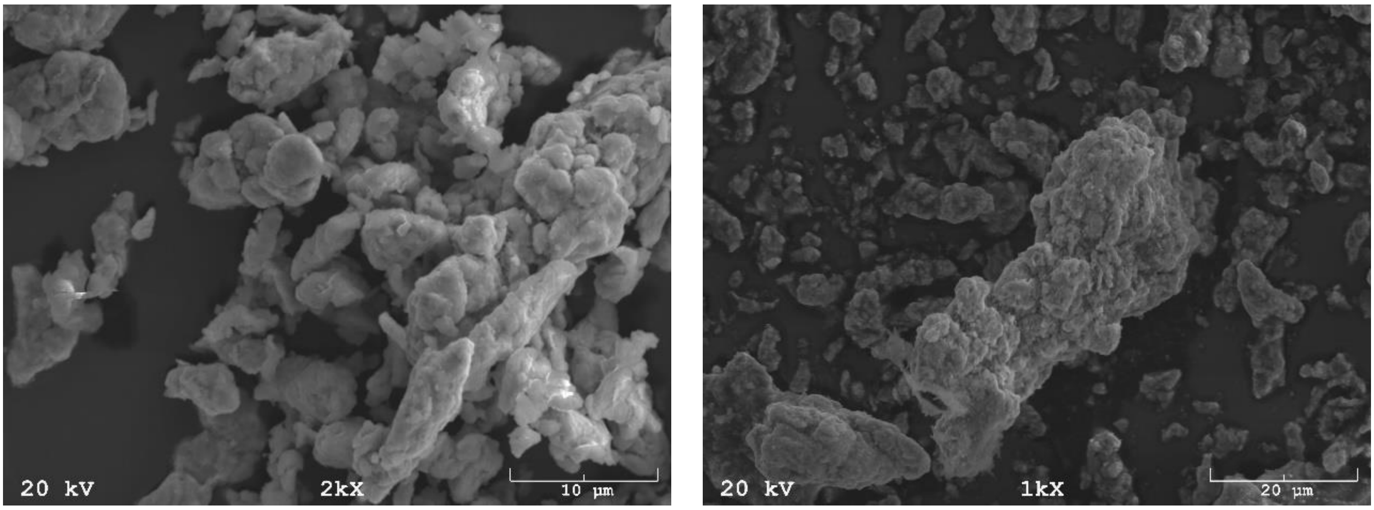
(**a**) (**b**)

Figure 2. Morphology of the particles of tuff used for research: (**a**) Tuff in the form of a fragment of a rock; (**b**) at a magnification of 3000X [33].



(**a**) (**b**)

Figure 3. Morphology of the particles of copper powder: (**a**) at a magnification of 500×; (**b**) at a magnification of 1000X [33].



(a) (b)

Figure 4. Morphology of the particles of copper with tuff after homogenization: (**a**) 5% wt. of tuff at a magnification of 2000×; (**b**) 30% wt. of tuff at a magnification of 1000X.

The morphology shows a tendency of the tuff particles to create agglomerations. This was taken into consideration during the mixing process; the planned mixing time was relatively long (12 h).

Additionally, thermogravimetric analyzes were made for the tuff to check its applicability for planned solutions and proper parameterization of the sample preparation. Figure 5 shows the changes in the tuff weight as a function of temperature.

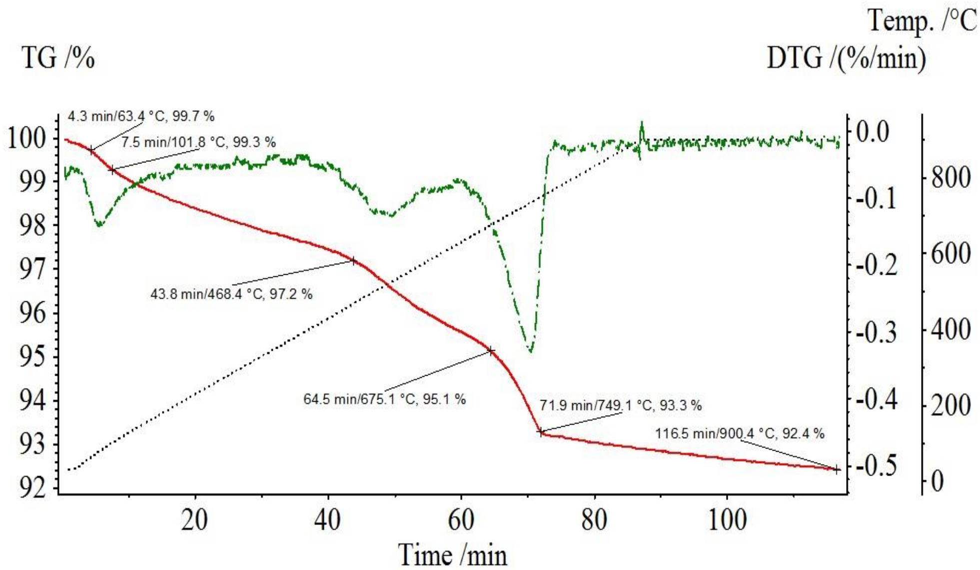


Figure 5. TG (red) and DTG (green) curves of the tuff with marked significant points.

It was observed that the tuff mass decreases with the temperature increases. At first, this effect is mainly related to the evaporation of moisture. Furthermore, water (m/z 17 and 18) is excreted from the sample in the temperature range from 30 °C to approximately 600 °C. Two maxima of water excretion are observed at the temperature of about 180 °C and the temperature of about 460 °C. In the first stage, the tuff is dehydrated, i.e., the absorbed water is removed, or possibly the inter-batch water is removed, while dehydroxylation is observed in the later stage. The thermal dissociation of calcite occurs at a temperature of about 740 °C. As a result of the reaction of decomposition of $CaCO_3$ compounds contained in the tuff, CO_2 (m/z 44) and C (m/z 12) emissions are observed. The stabilization of tuff mass takes place at temperatures above 740 °C.

As a matrix, the electrolytic copper powder was used (EUROMET, Trzebinia, Poland). The powder particles were characterized by dendritic shapes. The advantage of this shape is better homogenization, in comparison with spherical particles, during the mixing process. According to the producer's declaration, the powder includes 99.71% Cu. Other elements were: Pb, Fe, SO_4, O_2, and H_2O.

2.2. Sample Preparation

Preliminary research was carried out to select parameters for the production of copper compounds with volcanic tuff. They involve:

- density changes depending on the applied compaction pressure and sintering process parameters;
- determination of the relationship of the hardness after applied compaction pressure; and
- determination of the influence of the quality of copper powder and sintering atmosphere on the sintering process and the properties of the sinters.

Based on the results obtained in the preliminary research, the tuff composite manufacturing optimal parameters of the process of tuff composites manufacturing were determined due to the density of the composites obtained [33]. On this basis, the following process was designed.

Mixing of copper and tuff powders was carried out with the use of the Turbula rotary mixer for 24 h. Mixtures containing 5, 15, and 30% of the volume of the tuff were prepared. It was decided to introduce fine particles below 40 µm because the adhesion force between very fine particles and large particles is similar. In the study [34], the authors investigated the influence of the size of ceramic particles on the force of adhesion between the particles and the metal matrix. In the case of Cu-Al_2O_3 compounds with a 5% volumetric share of the ceramic phase, the particles were introduced in two variants: <3 µm and 180 µm coarse powder. It was noticed that the interface strength was higher for samples with large particles and amounts to 74 ± 4 MPa, while for fine particles it amounted to 68 ± 3 MPa. Information on the composition of particular samples is presented in Table 2.

Table 2. The samples designation.

Samples	Cu	Cu5T	Cu15T	Cu30T
Composition {%vol.}	100% Cu	95% Cu + 5% Tuff	85% Cu + 15% Tuff	70% Cu + 30% Tuff

The study was carried out on copper composite with tuff addition on cylindrical samples with a 20 diameter and 5 mm thickness for these tests. All samples were made by applying a 200 MPa one-sided compaction using a EU40 hydraulic press. To reduce the coefficient of friction between the powder and the matrix walls, the matrix walls were lubricated with zinc stearate.

The sintering process was carried out in a laboratory tubular furnace at a temperature of 900 °C in an atmosphere of nitrogen. Slow heating to isothermal sintering temperature was applied at a rate of 10 °C/min. The isothermal sintering time of the samples was 60 min. After sintering, the samples were cooled together with the furnace. To strengthen the samples after sintering, they were densified in a closed matrix at a pressure of 1200 MPa.

2.3. Methods

The density of the samples was determined using the geometric method. The dimensions of the samples were measured with a laboratory caliper with a measuring accuracy of 0.01 mm, and the weight of the samples was determined using the laboratory's precise analytical balance. Because the composites were not characterized by significant porosity (solid material without voids), the calculations were carried out for solid, non-porous materials.

Hardness tests were performed with the Rockwell method (B scale) according to EN ISO 6508-1:2007/Ap1:2009. Microhardness tests HV0.05 were performed on the FM 700E microhardness tester. The examination of the composite softening temperature was based on the examination of the hardness of samples after exposure at elevated temperatures (from 100 to 800 °C) for 2 h. The annealing processes were performed in a laboratory furnace in an air atmosphere. The softening temperature was defined according to ISO 5182:2008 as the maximum temperature that, if maintained for 2 h, will reduce the hardness of the material by 15% compared to the hardness of the material not subjected to annealing.

Electrical conductivity tests of the composites were performed with the use of a SIGMATEST device at The Welding Institute in Gliwice. The study consisted of measurement with an eddy current device that measures the electrical conductivity of non-ferrous metals on the basis of the impedance of a combined measurement probe.

After the basic tests, a series of trial electrodes was manufactured. The tests of electrode durability and weld quality were carried out. Powder metallurgy technology was used to produce electrode tips for spot welding. Cylindrical samples with dimensions of 25 × 35 mm were made. Then, by post-process machining, they were specially shaped and adapted to be used as caps for working parts of welding electrodes. A series of several hundred test welds were made on a typical 12 kVA spot welding machine. Evaluation of the durability of electrodes made of Cu-Tuff composites was performed on the basis of the EN ISO 8166 standard. The LogWeld measuring system was used to control the correctness of the welds performed.

3. Results

3.1. Density

The values of the density measurements are presented in Figure 6.

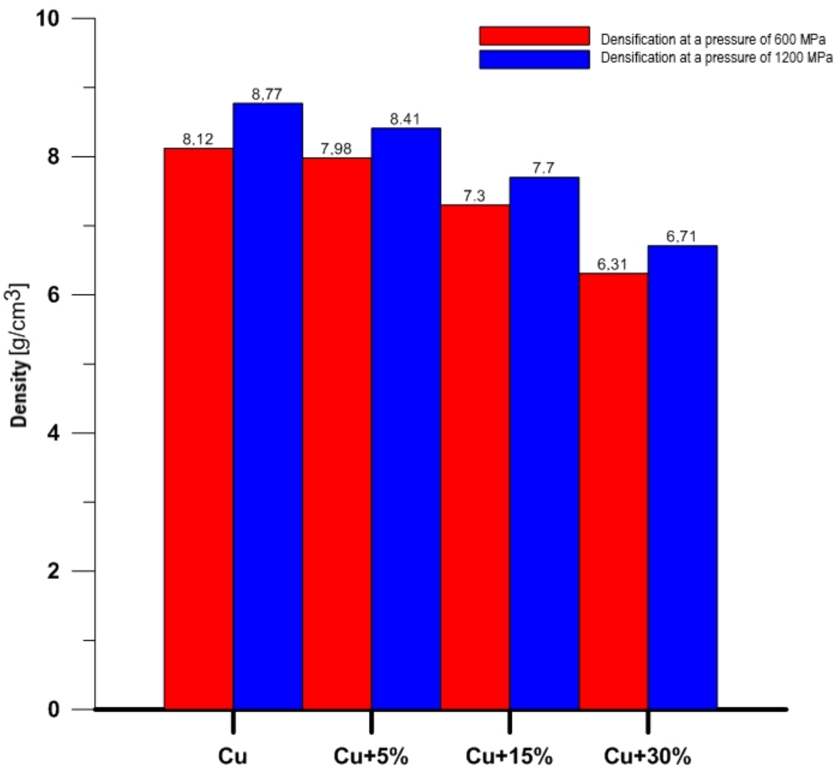


Figure 6. The densities after densification of pure copper and composites with tuff.

The results of the density measurements show higher values for higher pressure, as predicted. The best result was obtained for copper without the tuff admixture. The lowest values are for the 30% tuff admixture. The results show that the tuff caused a decrease in the density of the composite but led to an increase in relative density. The relative densities of the composites with tuff were: pure Cu = 86.9%; Cu + 5%= 94.1%; Cu + 15% = 96.8%; and Cu + 30% = 95.1%.

3.2. Hardness

The results of the hardness are presented in Figure 7.

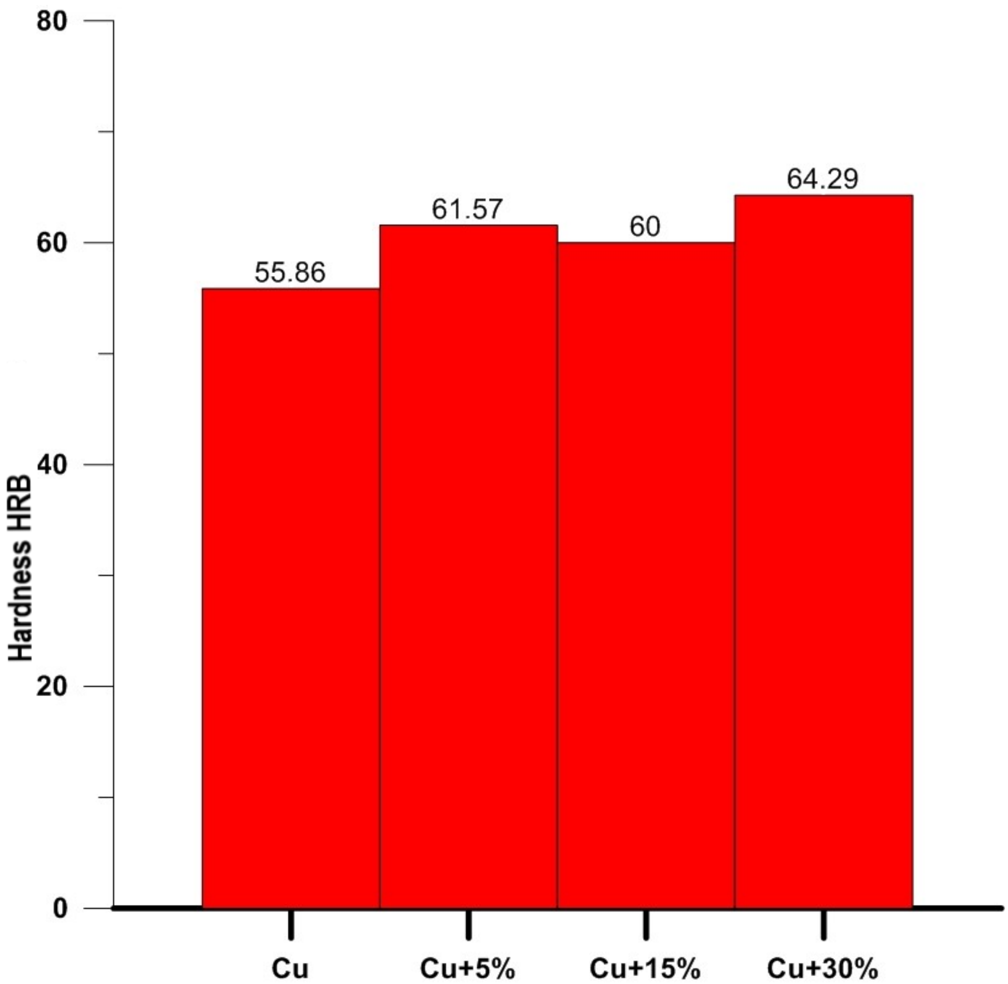


Figure 7. The hardness of composites with tufted densification in the matrix at a pressure of 1200 MPa.

The highest hardness values are obtained for the 30% tuff admixture; the lowest values for pure copper. The values for the 15% tuff admixtures and 5% tuff admixtures are quite similar, and the lowest values for the composites with 15% tuff admixtures are probably the effect of uneven particle distribution in the material value. The overall tendency shows that the tuff admixtures caused an increase in the hardness of the material.

3.3. Electrical Conductivity

The results of the electrical conductivity measurements are presented in Figure 8.

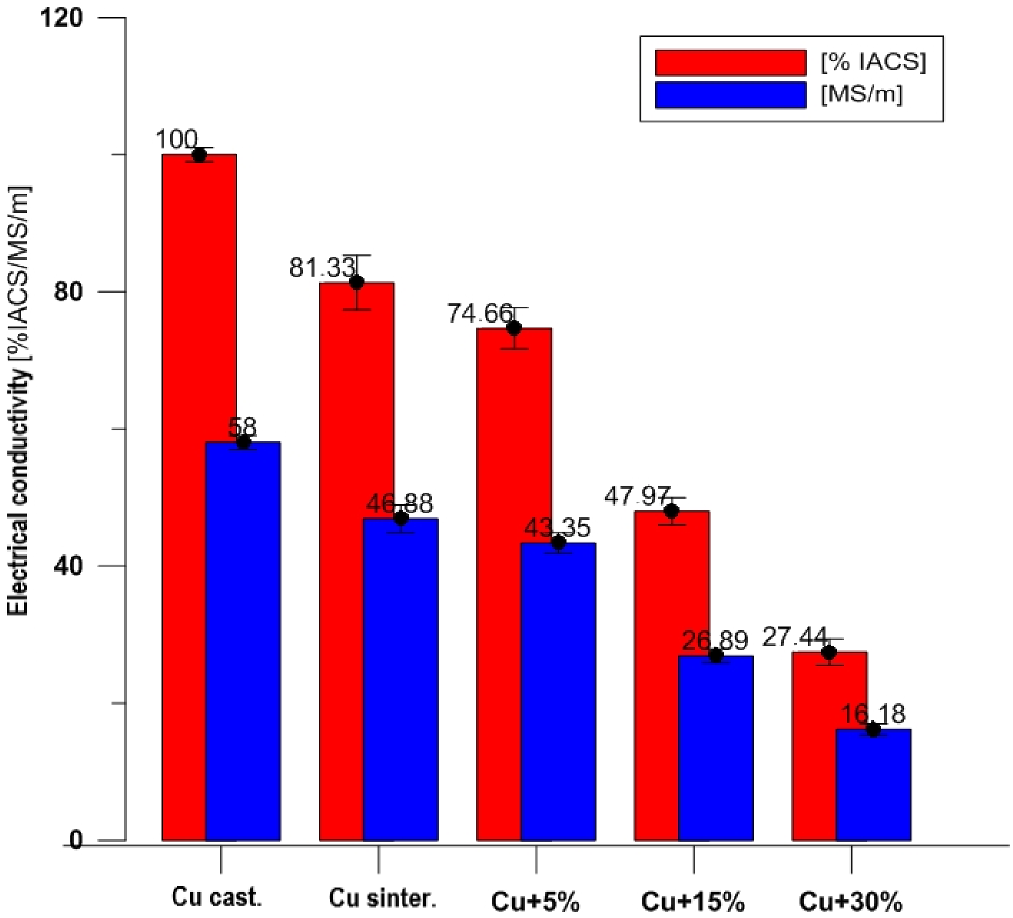


Figure 8. Electrical conductivity of compounds with tuff.

The results show the best electrical conductivity for a pure copper element made by the casting method; a lower value is obtained for the sintered copper element which is about 80% of the value of the casted material. Composites with tuff admixtures have lower values of electrical conductivity. However, the influence of the 5% tuff admixture is relatively lower than that of changing the method of production.

3.4. Comparison of Basic Parameters: Density, Hardness, and Electrical Conductivity

Basic parameters for all composites were measured and compared in Figure 9. Changes in the most important properties: softening temperature, relative density, electrical conductivity, and hardness of copper and Cu-Tuff composites are shown depending on the volume share of tuff particles.

When comparing the sinter of pure copper with the 5% tuff composite, one can see a very clear improvement in properties such as softening temperature, relative density, and hardness. However, as the content of the material increases, the conductivity of the composites decreases. This is a result of a decrease in the active cross-section of the composite due to the presence of non-conducting tuff particles. No diffusion of tuff components to copper or its contamination was found in the study conducted. The purity, and thus the conductivity of the matrix, remains unchanged. The electrical conductivity values of copper and tuff composites correspond to copper alloys used for resistance welding electrodes. The highest values of the relative density of composites were obtained for a tuff content of 15%. Compared to pure copper sinter, it is about a 10% increase in relative density.

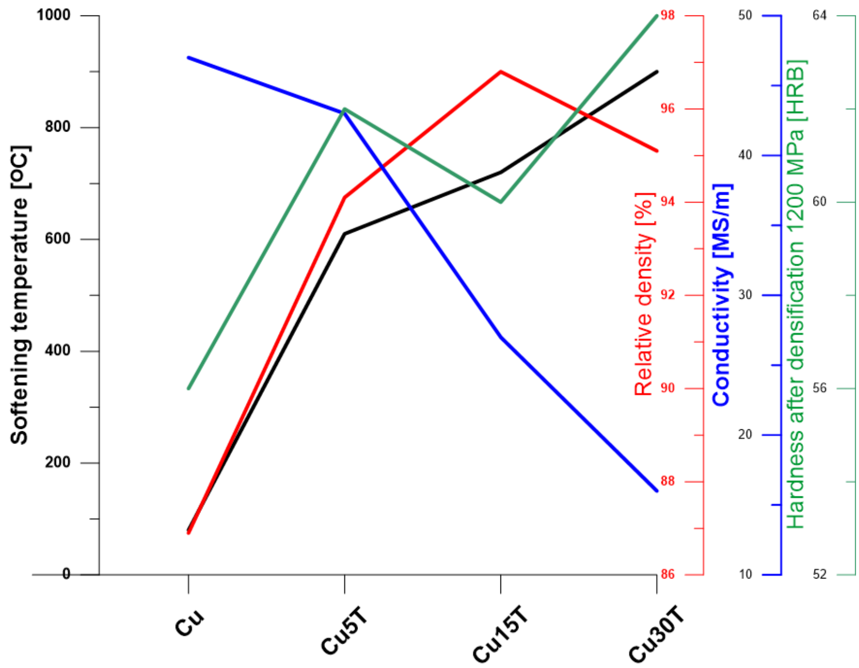
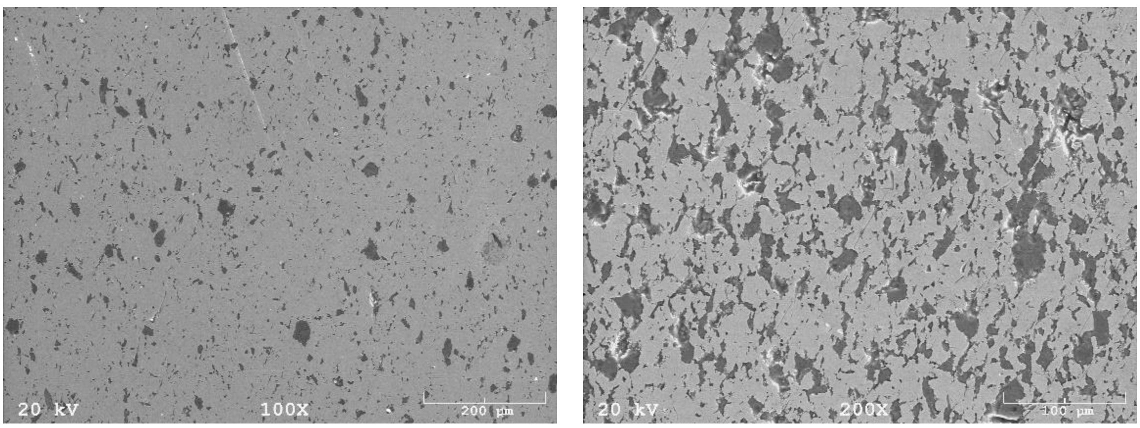


Figure 9. Effect of tuff content on the properties of copper matrix composites: softening temperature (black), relative density (red), electrical conductivity (blue), and hardness (green). Samples were compressed in the mold at 1200 MPa pressure.

The softening temperature of the composites studied increases with the increase in tuff amount. Already, 5% of the addition of tuff causes an increase in the softening temperature from 80 °C to more than 600 °C. Composites containing 30% tuff per volume have the highest softening temperature. They can work even at temperatures of about 900 °C. With the increase in the tuff volume share, the hardness of the composites increases after the re-pressing at 1200 MPa. Moreover, for a tuff content of 15%, a decrease in hardness is observed. This effect is caused by a smaller degree of composite deformation, which is connected with obtaining the highest value of relative density for this composition.

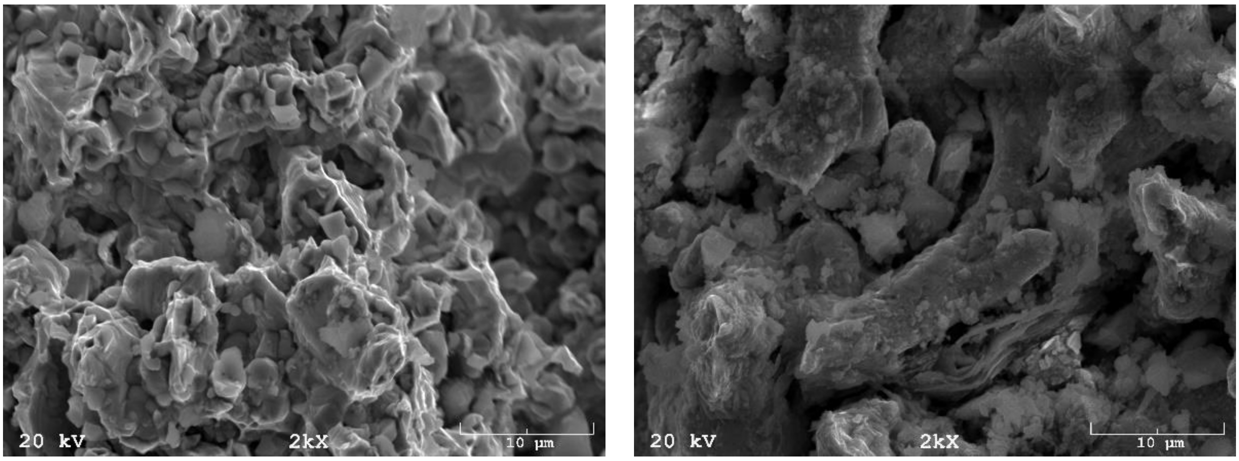
3.5. Microstructure

Tests of the microstructure were carried out by observing polished metallographic specimens (Figure 10) and by observing fractures of the samples (Figure 11).



(**a**) (**b**)

Figure 10. Microstructure of composites with: (**a**) 5% wt. of tuff at a magnification of 100×; (**b**) 30% wt. of tuff at a magnification of 200× [33].



(a) (b)

Figure 11. Microstructure of composites with: (**a**) 5% wt. of tuff at a magnification of 2000×; (**b**) 30% wt. of tuff at a magnification of 2000×.

The microscope investigation shows the even distribution of tuff particles in the copper matrix (Figure 10). It is important for a technical user to ensure the same material properties throughout the whole volume of electrodes.

SEM observation allows the confirmation of a good coherence of copper matrix and tuff particles (Figure 11).

3.6. Durability

The trial electrodes for durability tests were manufactured; some of them are presented in Figure 12. The electrode tip from the copper composite reinforced by 5% tuff by vol. with a 5 mm diameter and thickness of 7 mm were prepared. Next, they were soldered to standard copper mandrels.

(a) (b)

Figure 12. Socket electrode tips made of Cu5T copper composite and electrode pins on which the tips are mounted: (**a**) electrode tip; (**b**) electrode tip with the mandrel.

The durability was measured on a steel plate with dimensions of 470 × 350 mm. On each plate, 192 welds were performed (12 rows × 16 welds). Each electrode was used for 2000 performance welds. The dimensions were measured after every 100 welds. There was no increase in electrode diameter. The conducted tests did not show any electrode wear different from the commonly used alloys for resistance welding.

Additionally, the investigation of compressive strength for the welds was made according to EN ISO 14273. The results were compared with those of the traditional CuCrZr electrode and are shown in Table 3.

Table 3. Compressive strength test results.

Samples	CuCrZr after 1000 Cycles	Cu5T after 100 Cycles	Cu5T after 1000 Cycles
Compressive strength {MPa}	4.65	4.69	4.62

The conducted tests did not show significant differences between the new electrodes and the commercial one.

4. Discussion

Comparing pure copper sinter and a composite containing 5% tuff, a very clear improvement in properties such as softening point, relative density, and hardness after densification is visible. As the tuff content increases, the conductivity of the composites decreases. This is a result of the reduction in the cross-section of the composite due to the presence of non-conductive tuff particles. In the conducted tests, no diffusion of the tuff components into the copper and its subsequent contamination was found. The purity and thus the conductivity of the matrix remained unchanged. The decrease in conductivity was due to the presence of non-conductive ceramics. The electrical conductivity values of copper-tuff composites correspond to the copper alloys used for resistance welding electrodes. The highest values of the relative density of composites were obtained for the tuff content of 15%. However, these values are at a similar level for composites with 5%, 15%, and 30% tuff and they vary in the range of 94.1 to 96.8. Compared to pure copper sinter, this is an increase in relative density of about 10%.

Today, welding technology is one of the most widely used in industries such as aerospace, construction, automotive, and others. Technological development also requires new research for the application of these solutions for more demanding purposes, for example, underwater welding [35–38]. New materials solutions are also needed. Modern techniques such as 3D printing solutions [39] allow for the application of new and more advanced reinforcement in the copper matrix such as graphene admixtures or different nano-additives [40,41]. Not all of them are possible for application in the welding industry, but they show a modern trend in this area.

The presented research shows that volcanic tuff is an attractive reinforcing material for copper matrix composites for application in resistance welding electrodes. It could be a successful replacement for traditionally used materials as well as other, more expensive admixtures. It is also worth paying attention to the ecological nature of the proposed filler. In addition to being used in metal composites as reinforcement, it can be easily recycled. Composites reinforced with aluminosilicate sinter can be recycled through the spontaneous flow of liquid metal from the pores of the reinforcement. Recycling of such materials is possible as opposed to composites containing, e.g., graphite. The ceramic tuff particles in the metallurgical process float to the surface which makes it possible to recover practically pure copper [23,28].

5. Conclusions

The study carried out and presented in this paper demonstrated that volcanic tuff is an attractive reinforcing material for copper matrix composites. It significantly increases the softening temperature and additionally increases the resistance of composites to abrasion, while maintaining proper conductivity, meeting the standard requirements for resistance welding electrodes. As a result of the research, it was found that the addition of 5% tuff produces the best results in this type of composite. It was found that for the sample with a 5% share of tuff, a high softening point above 600 °C was obtained as well as a high hardness after densification at the level of 62 HRB, high relative density of approximately 95%, and very good conductivity at the level of approximately 45 MS/m.

Such a composite can be successfully used to produce cap electrodes for resistance welding machines. The use of this material will result in an increased resistance to the wear of electrodes, and the production process, as a result of the availability of tuff and its low cost, will not be expensive or complicated. Additionally, such composites can be successfully produced using powder metallurgy technology.

6. Patents

The solution presented consisting of the use of volcanic tuff in metal matrix composites is under patent protection by the Patent Office of the Republic of Poland, number PL 21781818—"The use of volcanic tuff to strengthen copper matrix sintered composites, copper matrix sintered composites reinforced with volcanic tuff particles, and the method of manufacturing copper matrix sintered composites reinforced with volcanic tuff particles".

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