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Copper-Graphene Composite Materials: Manufacturing Technology and Properties

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Abstract. The article presents the results of preliminary research into the feasibility of copper-graphene composite application in the production of shaped charge liners in HEAT ammunition from graphene-coated copper grain powders by powder metallurgy techniques. Copper powder grains were coated with graphene with a machine and process developed at the Institute of Precision Mechanics in Warsaw (Poland). The characteristics of the applied powdered materials (including particle size distribution) were determined in this work. The paper discloses the result of graphene identification by Raman spectroscopy.

The presence of graphene was confirmed in the processed copper powder. The paper discusses the preparation of copper powder by grinding (refinement) and reduction for consolidation. Powder mixtures of pure copper and graphene powder were applied at different component ratios. P/M compacts and sinters (agglomerates) for the test specimens were made from the proposed mixtures by die pressing and sintering in dissociated ammonia gas. Examples of microstructures and selected test results of material properties are shown for the produced sinters.

The paper shows a selection of test results for the copper-graphene composites produced by PPS (*Pulse Plasma Sintering*) from 100% graphene-coated copper powder. The properties of the produced composite materials were determined, including their density, porosity, and a selection of mechanical properties identified by DSI (*Depth Sensing Indentation*). It was found that the copper-graphene composite met the primary design criteria applied to shaped charged liners for HEAT munitions. In "traditional" powder metallurgy processes, high-density products can be produced if the composite material features a low content of graphene-coated copper powder; PPS, however, makes the production viable with 100% graphene-coated copper powder.
Keywords: powder metallurgy, shaped charge liners, graphene

1. INTRODUCTION

Shaped charge liners are components of Munroe-effect explosive systems which have been used for many years in various industrial sectors; primary examples include military ordinance (HEAT munitions) and civilian applications, like mine blasting works [1, 2]. Many research institutions have been investigating the feasibility of improving the effectiveness of shaped charge explosive systems. The research has resulted in HEAT munitions capable of piercing armour plating systems made from armour steel more than 6÷8 of the shaped charge explosive diameter [3]. The common shaped charge liner material is a range of copper grades of high purity – with over 99.95% of pure copper. Copper is the material of choice due to its good plastic properties which help form a high-velocity shaped charge jet from the liner. The level of contaminants, mostly oxygen, sulphur and phosphorus, of the copper material for shaped charge liners is reduced below 0.05%. The three elements form hard and brittle chemical compounds with copper [4–7], and significantly reduce the mechanical performance of the material [8].

A separate problem linked to ensuring the maximum possible plasticity of shaped charge liner materials is to achieve a specific material structure. The shaped charge jets formed from shaped charge liners, which are made from copper with an average structural grain size within 20–40 μm, had the best compliance with the design characteristics [9–11]. Increasing the structural grain size reduced the performance of the tested shaped charge explosive systems. Shaped charge liners made from materials with a fine-grain structure are also justified due to the uniform physical properties within the whole liner body.

The production of the designed and required fine-grain structure of pure metals is challenging. There are few crystal nuclei during solidification, and a small number of grains would easily grow. Tests and research are attempted to apply various manufacturing processes of shaped charge liners which would provide the required structures. Reference sources cite results from the research into alternative technologies for production of shaped charge liners. They include galvanic processes, plasma spraying and chemical vapour deposition (CVD). The yield of the processes is very modest. Hence, the cited research and tests should be regarded as being more educational than feasible for any application.

In the manufacturing practice of shaped charge liners from metallurgical materials, the material structure is refined by additional processing. Based on the research results published, for example in [12], there is a noticeably high interest in the application of plastic working combined with recrystallization annealing for structure refinement. The conditions and progress processes are well known and discussed in many publications, like [13].

The data from reference literature [5, 14-17] allowed a conclusion that powder metallurgy is a potentially viable manufacturing process of shaped charge liners. This is supported by the capability of powder metallurgy in production of:

- materials with the required chemical composition and purity;
- the resulting fine-grain structures and statistical uniformity of physical properties [9-11].

It is known that porosity is a characteristic feature of products made with powder metallurgy applications. The porosity levels depend on the specific manufacturing method applied [18]. Excessive porosity of a shaped charge liner material may result in 'powdery' shaped charge jets. References [3] specify that shaped charge jets formed from shaped charge liners made from sintered copper materials with a porosity share at or below 8% feature parameters approximate to those of solid-copper shaped charge liners.

The experimental tests discussed in this paper were carried out under a research project funded by the Polish National Centre for Research and Development (NCR&D). The objective of the project was to deliver laboratory test results which would confirm the effectiveness of innovative components of shaped charge explosive systems and enable the production of the systems in new types of HEAT ammunition, such as 40 mm rocket-propelled grenades.

The experiments carried out at Institute of Precision Mechanics (IPM, Warsaw, Poland) and Military University of Technology (MUT, Warsaw, Poland) were focused on ascertaining the feasibility of graphene as a component of material from which shaped charge liners would be produced.

Graphene is the latest form of engineered carbon compound physically produced at research laboratories [19].

Graphene is an allotropic form of carbon and can be fabricated in single layers, each of which is a two-dimensional structure. Graphene is a viable component of composite materials, like structural plastics or coatings [20]. In this work, copper powder coated with graphene and made at the IPM was used. The manufacturing technology of the material is protected by patent rights under Polish Patent PL.225890, "Sposób wytwarzania struktur węglowych zawierających grafen na proszkach miedzi z wykorzystaniem obróbki cieplno-chemicznej" (A method of production of carbon structures by chemical and heat treatment and with graphene-coated copper powder), dated 1 December 2016.

2. PREPARATION OF MATERIALS AND RESEARCH METHODOLOGY

To assess the feasibility of graphene-coated copper powder in the production of sinters (agglomerates), a methodology of specimen production had to be developed that was based on powder metallurgy technologies.

The first stage of material preparation for the research was to coat copper powder grains with a layer of graphene. This coating process was developed and completed by the IPM. The process consisted in the thermal treatment of the copper powder in a fluidized bed system and an atmosphere which contained the source of carbon.

Experimental tests were carried out to identify the deposited graphene by Raman spectroscopy. The characteristic Raman spectrum of the graphene present on copper grains is shown in Fig. 1.

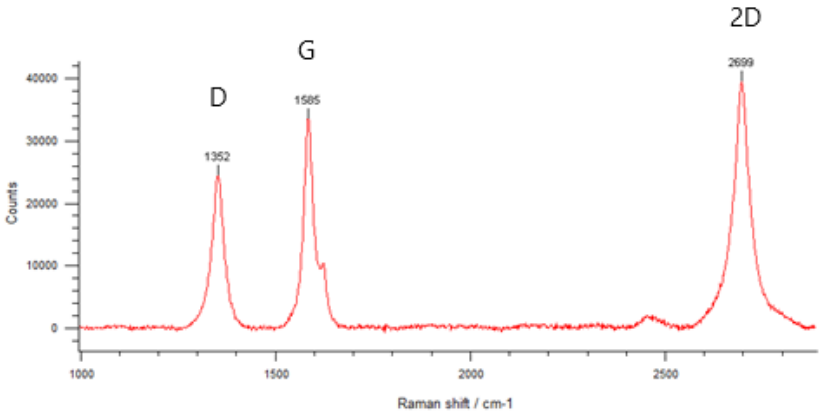


Fig. 1. Raman spectrum of graphene-coated copper powder

The Raman spectrum image (Fig. 1) featured peaks characteristic of graphene (D, G and 2D). The presence of the peaks confirmed that graphene was present in the copper powder (and in the P/M compact).

The intensity analysis of G and 2D peaks revealed that graphene was present in the form of several atomic layers.

The D peak present in the Raman spectrum image proved that the graphene layers were defective. The graphene-coated copper powders used in the research varied in the degree of defects and in the number of graphene layers. The manufacturing process of the sinters comprised the following stages in succession:

1. Preparation of powder mixtures;
2. Forming (die pressing);
3. Sintering;
4. Finishing of sinters to produce specimens for further testing.

The mixture of pure copper powder and graphene-coated copper grains was made in a paddle mixer. The mixing time was 1 h, which delivered a uniform mixture.

The results of prior tests revealed that the sinters made from Cu+CuG powder mixtures featured high areas made from graphene-coated copper powder only. This meant that the CuG powder was not refined during the preparation of the mixtures.

This required testing of a CuG powder refinement method which would allow the CuG powder to be added to the powder mixture. The following CuG powder refinement methods were tested:

1. Grinding by hand in a ceramic mortar, processing time 15 min (100% of CuG powder) — Specimen A;
2. Grinding in a planetary ball pulverizer with steel balls, processing time 1 h (100% of CuG powder) — Specimen B;
3. Grinding in a planetary ball pulverizer with steel balls, processing time 1 h (90% of CuG powder) — Specimen C;
4. Grinding in a high-speed mill, processing time 10 min (100% of CuG powder) — Specimen D;

The test results were referenced to unground CuG powder, or Specimen E. Figure 2 shows photographic examples of the powders following grinding with the various methods tested.

As shown in the SEM images, the grinding in the planetary ball pulverizer significantly flattened the powder grains. The powders ground in the high-speed mill were reformed into larger granules with a diameter exceeding 200 μm. The Specimen A grains retained an approximately dendritic shape.

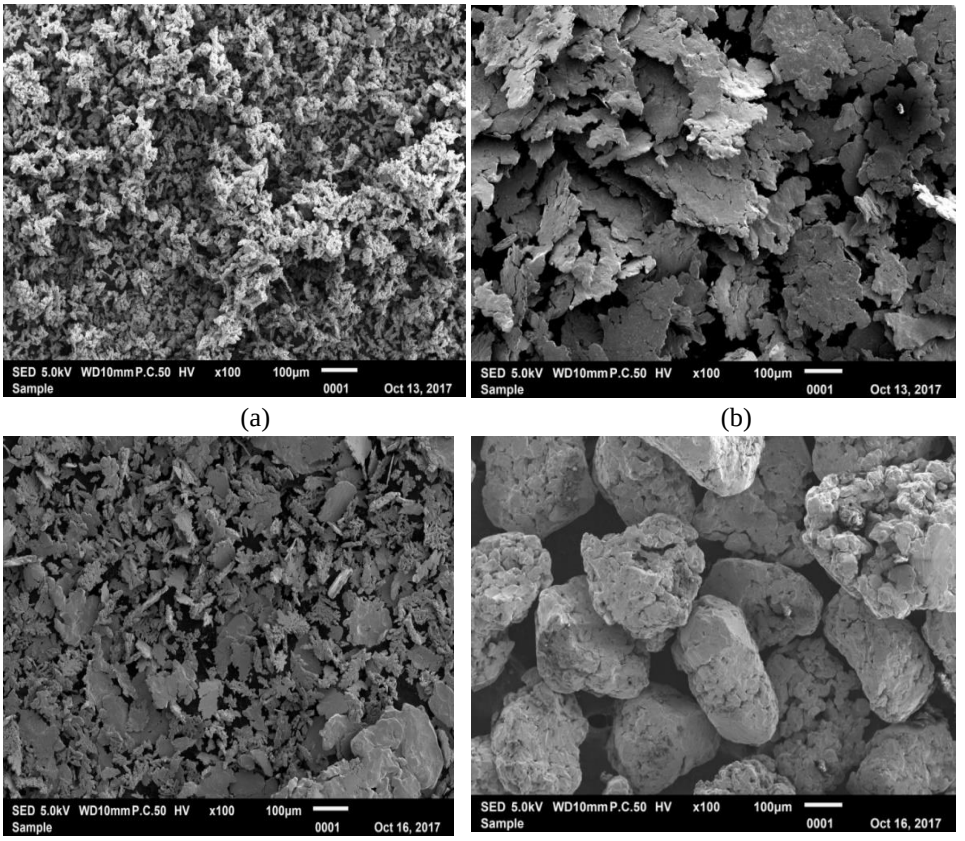


Fig. 2. SEM images of powders ground with various test methods: (a) – Specimen A; (b) – Specimen B; (c) – Specimen C; (d) – Specimen D (100x magnification)

The Raman spectroscopic assays revealed that the graphene layer was retained only in Specimen A after grinding. The grinding of the powder in the planetary ball pulverizer, the action of which had a very intense abrasion of the mixed material by the steel balls, could completely strip graphene off the surface of powdered copper grains.

Further into this research, the particle size of the tested powders was analysed by application of a Kamika solid particle grain-size tester.

Table 1 lists the assumed design diameter classes of the powder grains. Figure 3 shows an example of a mass particle size distribution of the powder grains. Table 2 shows the test results for the mean grain diameter of the powders produced by the different test grinding methods.

Table 1. List of assumed design diameter classes for powder grains

Class	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
Design grain	≤ 0.28	0.28 – 0.37	0.38 – 0.5	0.51 – 0.66	0.67 – 0.9	0.91 – 1.22	1.23 – 1.65	1.66 – 2.23	2.24 – 3.02	3.03 – 4.08	4.09 – 5.5	5.51 – 7.43	7.44 – 9.99	10 – 13.49	13.5 – 18.29	18.3 – 24.69	24.7 – 33.3	33.31 – 45.19	45.20 – 61.33	61.34 – 83.23	83.24 – 112.96

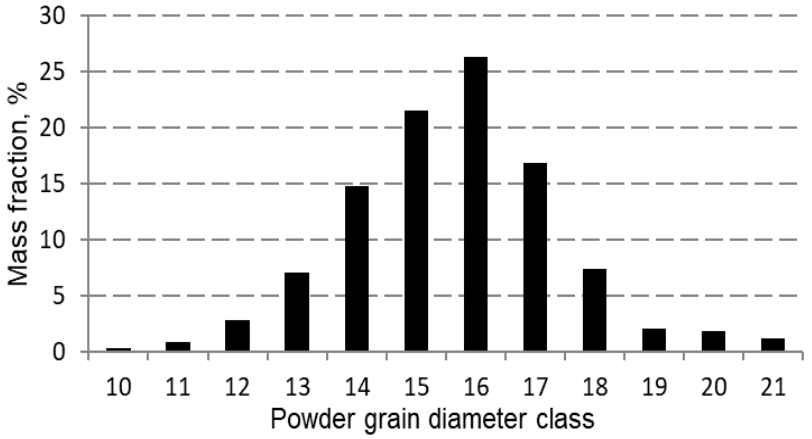


Fig. 3. Particle size distribution of Specimen A

Table 2. Test results of mean grain diameter

Specimen designation	A	B	C	D	E
Mean grain diameter of tested powders per mass distribution, μm	20.9	55.8	41.2	80.2	74.5

The results shown here indicate that grinding of the CuG powder in a planetary ball pulverizer with steel balls reduces the mean grain diameter; however, the irregular geometry of the grains (because of the grain flattening) could result in reduced compactibility. Specimen A underwent significant refinement without any flattening of the grains. The grains of Specimen A were found (by testing at IPM) with the graphene coating unscathed.

The grinding test method applied to Specimen A was qualified for further stages of the research work.

The next step in the production of the test specimens was die pressing at 300 MPa. The produced P/M compacts were sintered in a tubular furnace in an atmosphere of dissociated ammonia gas.

Reduction of the oxides was expected on the surfaces and inside the powder grains at the preliminary sintering stage. This required a temperature hold threshold with a defined duration at the stage of specimen heating to ensure that all related reactions occurred correctly.

To verify these assumptions, the reduction of copper powders was tested at 350°C and 550°C. The identical results proved that a reduction temperature of 350°C was enough for a full reduction of oxides. The test results are shown in Table 3.

Table 3. Loss of Cu powder mass following sintering in dissociated ammonia gas

Specimen no.	Reduced powder type	Loss of mass	
		Sintering temp. 350°C	Sintering temp. 500°C
1	Dendritic Cu powder, 40-63 μm	0.19	0.22
2	Graphene-coated dendritic Cu powder, 40-63 μm (IMP)	0.15	0.15
3	Dendritic Cu powder, 40-63 μm, reduced approx. 3 months before the test	0.61	0.61

For most samples, full reduction was achieved already at 350°C. It could be assumed that further holding of the P/M compacts for a long time above the temperature hold threshold will permit a full reduction of oxides. Increasing the temperature value anywhere above the tested temperature hold threshold would be unfavourable: rapid heating of a tested P/M compact might cause the pores to close before the oxides are fully reduced. The premature sealing of the pores could result in hydrogen disease.

The main sintering process parameters were first adopted from reference sources: temperature of 920°C for 1 hour.

The tests included in parallel the other method of graphene-copper composite manufacturing: PPS (*Pulse Plasma Sintering*). PPS was only applied to sinter the CuG (graphene-coated copper) powder with a grain size of 40-63 μm. The PPS method was characteristic in that following the preliminary die pressing at 200 MPa, the resulting P/M compact was pressed during sintering. The PPS pressure was tested at 70 MPa.

The specimens manufactured with the PPS method had a form of tablets sized ø20x5 mm. The PPS method was also used to make sinters of pure copper (Cu) powder, for comparative analysis.

For the strength and metallographic testing, the following specimens were made:

1. M1E solid copper specimens;
2. M1E solid copper specimens; heat treated during graphene coating;
3. M1E solid copper specimens, with the grain surfaces coated with a layer of graphene;
4. Sinters made from ECu1/0.040 - 0.063 μm powder;
5. Sinters made from ECu1/0.040 - 0.063 μm, with the grain surfaces coated with a layer of graphene;
6. Sinters made from ECu1/0.040 - 0.063 μm, with the grains coated with a layer of graphene (by PPS);
7. Sinters made from a mixture of 5%CuG powder and 95%Cu powder;
8. Sinters made from a mixture of 10%CuG powder and 90%Cu powder.

A part of the specimens made from M1E solid copper and sintered from ECu1/0.040 - 0.063μm had a layer of graphene deposited to the grain surfaces. Another part was placed in the graphene coating furnace (built by IPM) and only passed the heat treatment applied during graphene coating (this was done without a chemically active atmosphere, which was replaced with an inert gas shield). This process was intended to prepare the specimens for testing of the effect of graphene deposited on outer parts of materials on their mechanical properties.

The metallographic specimens were made by cutting the sinters across with a disc cut-off machine, followed by power grinding with a Struers Planopol 3 buffing grinder and sand-paper grit numbers from # 80 to # 4000, and finished by buffing with a diamond emulsion graded 3 and 0.25 μm in succession.

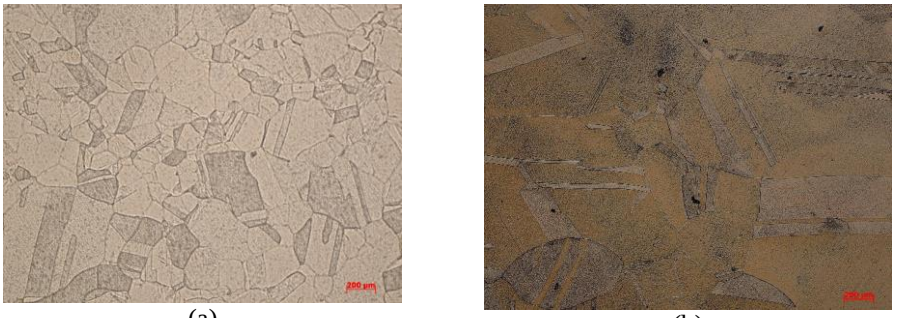
The microstructure of the tested sinter specimens and shaped charge liners was developed chemically with a Kalling's etchant. The microscopic examination of etched fractures was done with a Zeiss Axio Observer light microscope. The grain size was determined in Image J software. 10 photographs of the microstructure were analysed per sample. A median filter was applied to the image to improve its quality. The image was segmented with a double thresholding method.

The compressive and tensile test specimens were fabricated according to the applicable Polish Standard and tested with an MTS machine. Hardness tests were done with a Brinell machine.

3. TEST RESULTS AND ANALYSIS THEREOF

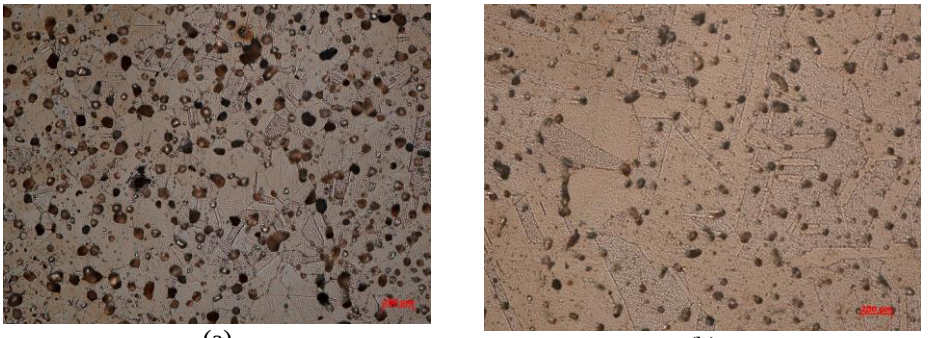
The research work involved test sintering of materials with a defined chemical composition, followed by the determination of their primary mechanical properties and microstructural analysis.

Figures 4 and 5 show the images of the examples of test specimens made from solid coppers and powders, both prior to and after depositing a layer of graphene on the grain surfaces.



(a) (b)

Fig. 4. Microstructure of (a) a solid Cu specimen and (b) a solid CuG specimen (5x magnification)



(a) (b)

Fig. 5. Microstructure of (a) a Cu 40-63 μm powder specimen and (b) a solid CuG specimen (50x magnification)

In both illustrated cases, the deposition of the graphene layer caused a significant growth of the grains. The results of the observations were confirmed by the test results for the mean grain size of the microstructure (Table 4).

A significant growth of the grains in the superficially graphene-coated specimens was identified too. The specimens made from sintered Cu and CuG powders had a small increment in the grain size. It was noticed that an increase of the CuG powder fraction in the mixture reduced the mean grain size of finished sinters. Table 5 shows the results for selected strength properties from tensile testing, hardness testing and density testing. The specimens tested featured varying shares of CuG powder. Other specimens tested included graphene-coated grains only and those heat treated in a process applied for graphene coating.

All the specimens were made from the powders by die pressing, followed by sintering in a dissociated NH_3 gas atmosphere. The test results were analysed as follows in this paper.

Table 4. Mean grain size for initial sinters

Specimen designation	**Mean grain size, μm**
Solid copper	65
Specimen made from sintered Cu 40-63 μm powder	16
Solid copper, heat treated	1150
Specimen made from sintered Cu 40-63 μm powder, heat treated	194
Solid copper, superficially coated with graphene	880
Specimen made from sintered Cu 40-63 μm powder, superficially coated with graphene	59
Sintered 95Cu+5CuG	20
Sintered 90Cu+10CuG	13

The density and relative density tests done on the sinters made by PPS revealed that the PPS method was viable to produce sinters from CuG powders (Fig. 6). The results of sintering the CuG (graphene-coated copper) powder and pure Cu shown in Fig. 5 suggested that it might be feasible to produce sinters from CuG powder with a density much higher than possible in traditional sintering processes.

The density of a sinter depends on the sintering temperature: the higher the sintering temperature, the higher the sinter density is.

The best results were achieved at a sintering temperature of 900°C. The porosity rate of the sinters was between 3% and 9%. The test results met the requirements specified for shaped charge liners produces in PM processes.

The mechanical properties of the PPS sinters (formed by sintering at 900°C) were DSI-tested (by *Depth Sensing Indentation*). DSI enabled determination of the hardness values (H_{IT}) determinable with more "traditional" tests, and determination of Young's modulus and Martens' hardness (H_M), where the latter includes plastic and elastic strains.

A DSI test is made by analysing the progress of penetration of a test specimen by an indenter, where the indenter load force and displacement during plastic and elastic strain are recorded. The results of these test are listed in Table 6 and shown in Fig. 6.

Table 5. Cumulative test results for sintered P/M and solid copper specimens

Item	Specimen designation	Yield point [MPa]	Elongation, [%]	Ultimate tensile strength, [MPa]	Density [g/cm^3]	Hardness, HB 2.5/15.625
1	Solid copper	277	12.4	280	8.92	88.6
2	Specimen made from sintered Cu 40-63 µm powder	77.1	9.1	168	8.07	46.5
3	Solid copper, heat treated	22.9	23.0	235	8.92	28.8
4	Specimen made from sintered Cu 40-63 µm powder, heat treated	40.0	19	173	8.32	23.2
5	Solid copper, superficially coated with graphene	21.4	27.0	237	8.92	27.6
6	Specimen made from sintered Cu 40-63 µm powder, superficially coated with graphene	37.0	16.4	177	8.31	35.7
7	Sintered 95Cu+5CuG	84.5	6.3	148	8.24	54.3
8	Sintered 90Cu+10CuG	84.0	4.0	124	8.08	52.4

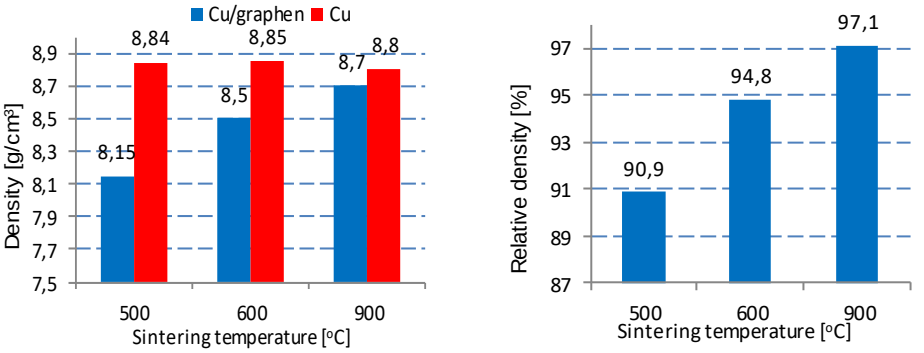


Fig. 6. Density of sintered specimens made from CuG+Cu and compaction of sintered CuG specimens

Table 6. DSI test results for the PPS graphene-copper composite material

CuG composite condition	**Load P_{max} [mN]**	**Loading rate, V [mN/min]**	**Young's modulus [GPa]**	**H_{IT} [MPa]**	**H_M [MPa]**	**Hardness [HV]**
P/M compact	500	500	48.1	327.8	270.1	32.9
Sinter 900°C	500	500	98.0	379.3	341.7	35.5

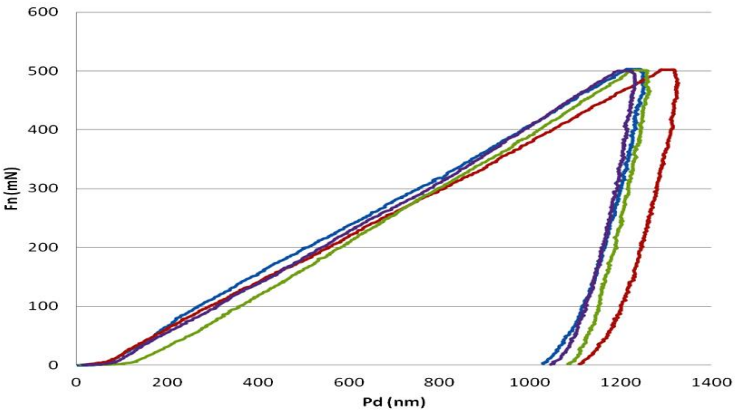


Fig. 7. Plot of the loading force vs. indentation depth curves determined by DSI testing

The sinter properties, hardness and Young's modulus were like the properties of as-sintered solid copper. A conclusion is valid that the CuG composite featured extremely good plastic properties and good elastic properties, comparable to those of solid copper.

4. SUMMARY OF RESULTS AND CONCLUSIONS

Figures 8 and 9 show the comparative test results for selected mechanical properties of the sintered specimens. The tests carried out on the M1E solid copper specimens and the specimens of sintered mixtures of Cu 40 - 63μm and CuG powder revealed that the addition of CuG powder increased the yield point and hardness limits, while reducing tensile strength and plastic properties (elongation) of the sintered specimens.

Further experimental tests were carried out to determine the effect of coating the outer surfaces of test materials with graphene on selected mechanical properties of the materials.

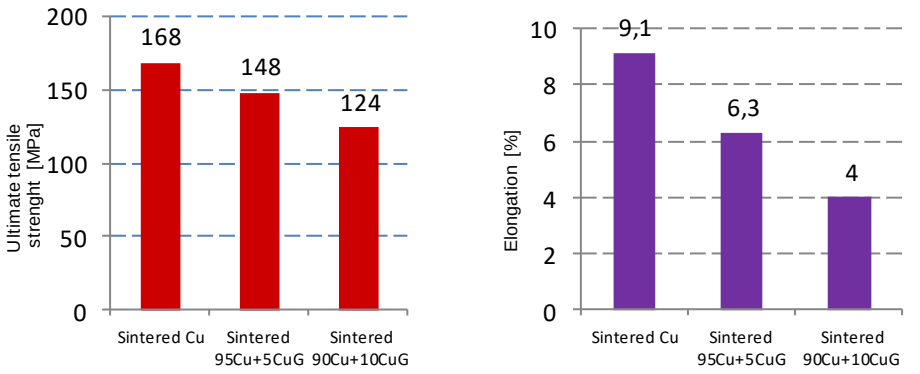


Fig. 8. Effect of graphene content on (left) tensile strength and (right) elongation value

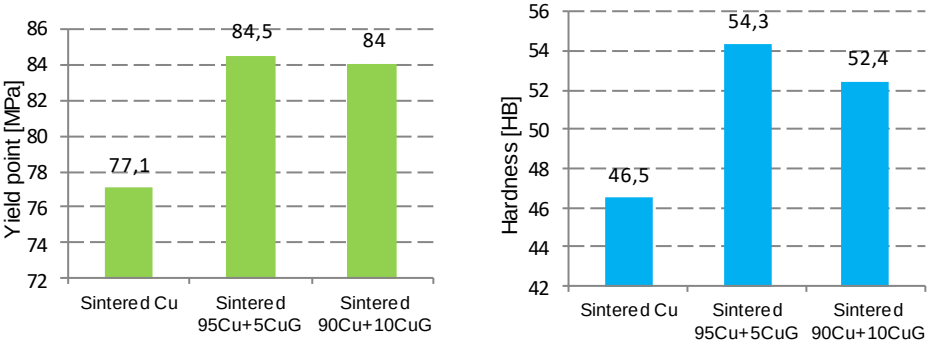


Fig. 9. Effect of the graphene content on (left) the yield point and (right) hardness of sintered specimens

The heat treatment processes as applied in graphene coating helped test the relationship while excluding changes in properties caused solely by the thermal processes. The test results are shown in Fig. 10 and 11.

As the test results suggest, the graphene coating reduced the strength properties (i.e. tensile strength, hardness, and especially yield point) while increasing plastic properties (elongation). The relationships occurred both in solid copper specimens and sintered copper specimens, although the latter had no significant change of tensile strength caused by heat treatment and graphene coating.

The conclusions from these experimental tests are generally consistent with the conclusions derived by microstructural testing of the specimens made from solid copper and sintered copper powders. A significant growth of grain size was discovered, which resulted in a lower yield point (according to the Hall-Petch law) with an increase of elongation.

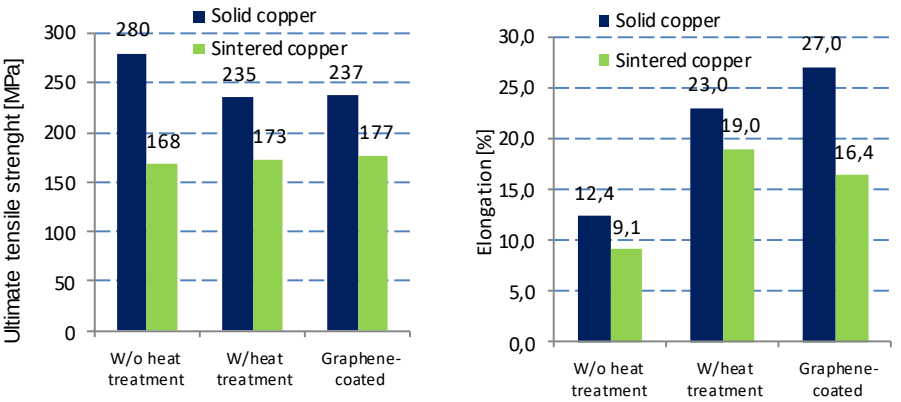


Fig. 10. Effect of the material type and graphene coating on (left) tensile strength and (right) elongation value

In most cases tested, the properties of solid copper were higher than in sintered copper; with the only exception being yield point.

The testing of the effect of heat treatment only on the properties of the materials led to a conclusion that in most cases it was the heat treatment (and not the graphene coating) that contributed to the changes in the properties. This led to a conclusion that coating the specimen grains with graphene was unreasonable and the same results could be achieved with a suitable heat treatment process alone. It could be only observed that the graphene coating process slightly improved the plastic properties of solid copper only.

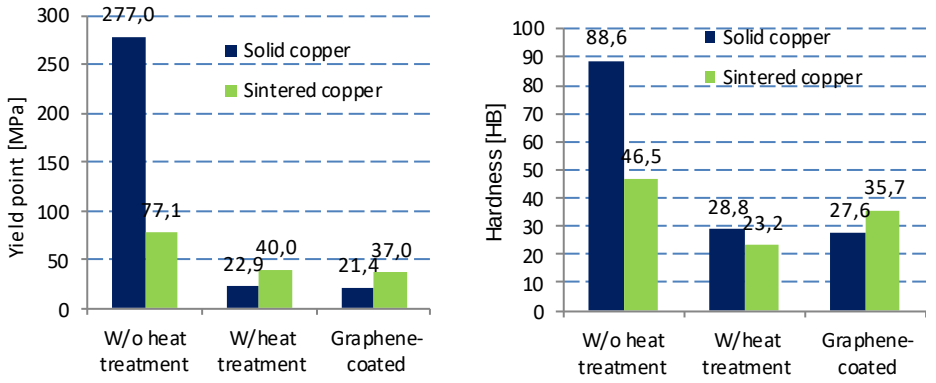


Fig. 11. Effect of the material type and graphene coating on (left) yield point and (right) hardness of the sintered specimens

It seemed, however, that the use of shaped charge liners made from CuG powders in further research will not deliver the desired result (due to a very high growth of grain size), which is an improved armour piercing (penetration) capability.

Further investigation should facilitate a determination of the properties of researched materials exposed to dynamic loads. This can help improve the assessment of the feasibility of application in shaped charge liners.

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Kompozyty miedź-grafen: technologia i właściwości

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Streszczenie. W artykule przedstawiono wyniki wstępnych badań dotyczących możliwości zastosowania kompozytu miedź-grafen, jako materiału przeznaczonego do wytwarzania wkładek kumulacyjnych amunicji przeciwpancernej technologią metalurgii proszków z proszków miedzi pokrytych grafenem. Grafenowanie proszków realizowane było w oparciu o urządzenie i technologię opracowaną w Instytucie Mechaniki Precyzyjnej w Warszawie.

W pracy określono charakterystyki zastosowanych proszków (np. rozkład granulometryczny) oraz pokazano wyniki dotyczące identyfikacji grafenu na podstawie analiz wykonanych metodą spektroskopii Ramanowskiej. Potwierdzono obecność grafenu na proszkach miedzi. Przedstawiono informacje dotyczące przygotowania proszku do procesu konsolidacji, obejmujące jego rozdrabnianie i redukcję. Zastosowano mieszanki proszków czystej miedzi z proszkiem grafenowanym o różnych proporcjach. W celu wytworzenia wyprasek i spieków (w postaci próbek badawczych) z zaproponowanych składów mieszanek zastosowano prasowanie matrycowe oraz spiekanie w atmosferze zdysocjowanego amoniaku. Przedstawiono przykładowe obrazy mikrostruktury oraz wybrane wyniki badań właściwości otrzymanych spieków.

W pracy przedstawiono również wybrane wyniki badań kompozytów miedź-grafen wytwarzanych metodą PPS (*Plasma Puls Sintering*) z grafenowanego proszku miedzi (zaw. 100%). Określono podstawowe właściwości otrzymanych kompozytów, tj. m.in. gęstość, porowatość oraz wybrane właściwości mechaniczne wyznaczone metodą DSI (*Depth Sensing Indentation*).

Stwierdzono, że kompozyt miedź-grafen spełnia główne wymagania stawiane materiałowi na wkładki kumulacyjne. W przypadku „tradycyjnych" technologii metalurgii proszków uzyskanie wyrobu o wysokiej gęstości możliwe jest w przypadku kompozytu o ograniczonej zawartości grafenowanego proszku miedzi, natomiast dla technologii PPS również dla 100% zawartości proszku grafenowanego.

Słowa kluczowe: metalurgia proszków, wkładki kumulacyjne, grafen