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Properties of Cu-based Alloys-powders for Brazing Prepared by Water Atomization

Lastnosti prahov-zlitin na osnovi bakra izdelanih z vodno atomizacijo

B. Šuštaršič*, B. Breskvar, V. Leskovšek, A. Rodič, Institute of Metals and Technologies, Ljubljana, Slovenia

Cu based alloys-powders are currently used as solder and brazing agents in many fields of application. Cu-Ag based alloys-powders are the most common brazing agents used in electrical engineering and electronics. Similarly, Au-Cu based alloys-powders find many applications in electrical engineering and electronics as well as in jewellery. Vacuum brazing of tool and high speed steels as well as hard metals (cemented carbides) on the structural steel base with Cu-Ni or Cu-Cr base alloys-powders is also increasingly used in many fields of application. Fluid and centrifugal atomization are the most frequent and therefore the most important manufacturing methods for the production of Cu based alloys-powders. Among the fluid atomization processes, gas and water atomization are the most common and the most important ones. The stream of liquid metal is disintegrated by high pressure jets of inert gas (N_2, Ar or He) or water. Gas atomization makes it possible to produce high quality, on the surface non-oxidized spherical powder particles. Water atomization is a simpler and cheaper powder manufacturing method. The metal powder produced by water atomization usually has irregularly shaped particles which are coated with a thin oxide film. In spite of that, the gas atomized powders could be replaced by water atomized powders in many fields of application. We therefore investigated the applicability of water atomization for the preparation of Cu based alloys-powders. The present article introduces the morphological and microstructural characteristics of prepared Cu-based alloys-powders and possibilities for their application.

Key words: powder manufacturing, powders for brazing, water atomization, properties of Cu-based alloys-powders.

Prahovi-zlitine na osnovi bakra se uporabljajo na mnogih področjih kot sredstva za mehko in trdo spajkanje. V elektrotehniki in elektroniki so najpogostejše spajke na osnovi Cu-Ag. Podobno se zlitine na osnovi Au-Cu uporabljajo v elektrotehniki, elektroniki in tudi zlatarstvu. Vakuumsko spajkanje orodnih jekel ali karbidnih trdin na osnovo iz konstrukcijskega jekla s prahovi-zlitinami na osnovi Cu-Ni ali Cu-Cr se tudi vse bolj uveljavlja v praksi. Prahovi-zlitine na osnovi Cu se najpogosteje izdelujejo s tekočinsko in centrifugalno atomizacijo. Med postopki tekočinske atomizacije se najpogosteje uporabljata plinska in vodna atomizacija. Curek raztaljene kovine pri teh postopkih razpršimo v hitro strjene delce prahu s pomočjo visokotlačnih curkov inertnega plina (He, Ar ali N_2) ali vode. Plinska atomizacija omogoča izdelavo visoko kakovostnih neoksidiranih prahov kroglične oblike. Vodna atomizacija je enostavnejši in cenejši postopek, vendar so tako izdelani kovinski prahovi običajno površinsko oksidirani in imajo zato tudi nepravilno oblikovane delce. Kljub temu bi lahko vodno atomizirani prahovi na mnogih področjih zamenjali dražje plinsko atomizirane prahove. Zato smo raziskovali možnost uporabe vodne atomizacije za izdelavo kovinskih prahov-spajk na osnovi Cu. V pričujočem delu so predstavljene morfološke in mikrostrukturne značilnosti izdelanih prahov ter posledično možnosti njihove praktične uporabe.

Ključne besede: izdelava kovinskih prahov, prahovi za trdo spajkanje, vodna atomizacija, lastnosti prahov zlitin na osnovi Cu.

1. Introduction

Metal powders are used extensively as filler material in the brazing and soldering industries[1]. Powders offer the most convenient method of applying filler metal to parts which have to be joined (bonded) together, although alternative filler metal forms (wire, rod, sheet, foil, etc.) are also used. Filler metals (alloys) for brazing have liquidus temperature above 450°C and below the solidus of the base metal, and filler metals for soldering have liquidus temperature below 450°C and below the solidus of the base metal. For brazing and soldering applications metal alloys-powders either as pure powder without additions or in flux-powder paste form are used.

Cu based alloys-powders are currently used as brazing agents (filler material) in many fields of application. Cu-Ag based alloys-powders are the most common brazing agents used in electrical engineering and electronics. Similarly, Au-Cu based alloys-powders find many applications in electrical engineering and electronics as well as in jewellery. Vacuum brazing of tool and high speed steels (HSS) as well as hard metals (cemented carbides) on the structural steel base with Cu-Ni or Cu-Cr as well

* Mag. Borivoj ŠUŠTARŠIČ, dipl. inž.,
IMT Ljubljana, Lepi pot 11, 61000 Ljubljana

as Ni-Cr base alloys-powders is also increasingly used in many fields of application[2,3].

Fluid and centrifugal atomization are the most frequent and therefore the most important manufacturing methods for the production of Cu based alloys-powders. Among the fluid atomization processes, gas and water atomization are the most common and the most important ones[4,5]. The stream of liquid metal is disintegrated either by high pressure (commonly 10 - 30 bars) jets of inert gas (N_2, Ar or He) or by high pressure (commonly 100 - 300 bars) jets of water. **Fig. 1** represents schematically a gas atomizer and the disintegration of a free falling molten metal stream by high pressure jets of inert gas.

Gas atomization makes it possible to produce high quality, on the surface non-oxidized spherical powder particles. Water atomization is a simpler and cheaper powder manufacturing method. **Fig. 2** shows schematically the production of metal powder by water atomization.

The metal powder produced by water atomization usually has irregularly shaped particles which are coated with a thin oxide film. In spite of that, the gas atomized powders could be replaced by water atomized powders in many fields of application. We therefore investigated the applicability of water atomization for the preparation of Cu based alloys-powders.

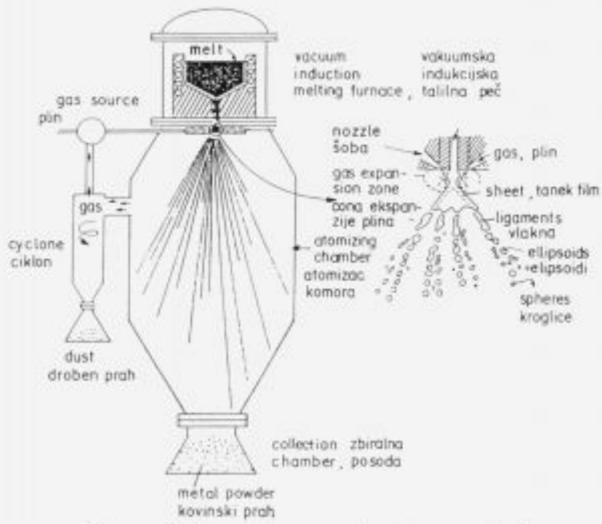

Figure 1: Schematic presentation of gas atomizer[4]

Slika 1: Shematični prikaz postopka plinske atomizacije[4]

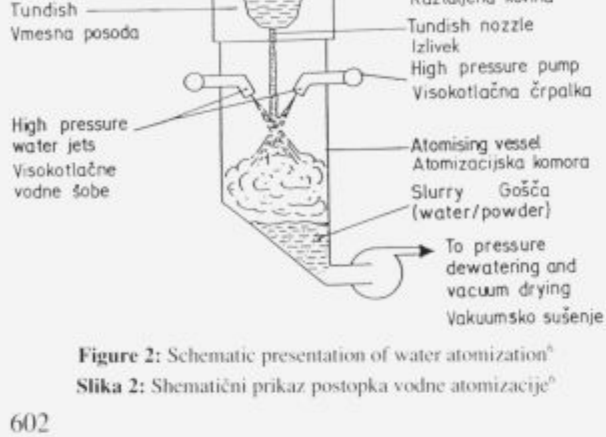

Figure 2: Schematic presentation of water atomization[6]

Slika 2: Shematični prikaz postopka vodne atomizacije[6]

2 Experimental procedure

Most of the experiments and investigations have been done at the Institute of Metals and Technologies, Ljubljana.

Two different vacuum brazing alloys (Cu with 2% of Ni and Cu with 1.5% of Cr) and two typical Cu-Ag based brazing alloys (L-Ag15P and L-Ag40Cd) were selected for our experiments and investigations. For the comparison some other types of alloys-powders (Ni and Co-based powders for welding, HSS powders, Alnico hard magnetic powders) of previous investigations[7,8,9,10] were also used. The powders of selected alloys were prepared by water atomization (pilot atomizer Davy McKee, type D5/2) installed at IMT Ljubljana (see **Fig. 3**).

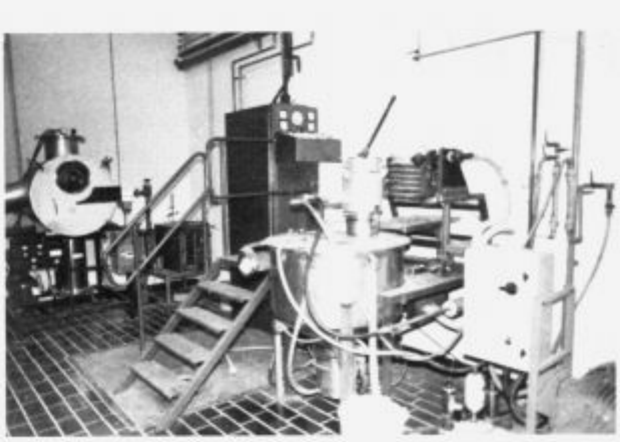

Figure 3: PM&RST laboratory at the IMT Ljubljana with water atomizer in the foreground.

Slika 3: PM&RST laboratorij na IMT Ljubljana z vodnim atomizerjem v ospredju.

Table 1: Chemical compositions of prepared Cu-Ag based water atomized powders in comparison with ASTM standardized[1] brazing alloys-powders.

Tabela 1: Kemične sestave z vodno atomizacijo pripravljenih prahov na osnovi Cu-Ag v primerjavi z zlitinami standardiziranimi po ASTM standardih[1]

Material	B CuP-5	L Ag15P	B Ag-2	L Ag40 Cd
	nominal	IMT	nominal	IMT
Elements	(mass %)			
Cu	≥79,85	balance	26,0	19,0
Ag	15,00	14,9-15,60	35,0	40,0
P	5,00	6,0-6,20	–	–
Zn	–	–	21,0	21,0
Cd	–	–	18,0	20,0
Other elements	0,15max.	not determined	0,15max.	not determined

Table 2: Process parameters of water atomization for the preparation of Cu-2%Ni and Cu-Ag based alloys-powders.

Tabela 2: Procesni parametri postopka vodne atomizacije za pripravo kovinskih prahov Cu-2%Ni in Cu-Ag.

Process parameter (water atomizer D5/2 Davy McKee	Cu-2%Ni alloy	Cu-Ag alloys	Remarks
Temperature of superheating (°)	1230±20°C	810±30°C	pyrometer
Tundish temperature (°)	1210±20°C	850±30°C	Pt-PtRh 13
Tundish nozzle diameter (mm)	∅4,0/4,5	∅4,0/4,5	fused quartz
Metal flow rate (kg/min.)	4,5-5,5	5,6-7,3	
Water nozzle diameter (mm) two (2) main nozzles two (2) side nozzles	 1,2 x 1,05 1,1 x 0,85	 1,2 x 1,05 1,1 x 0,85	 1503 type 1502 type
Jet apex angle (°) between main water streams between side water streams	 50 40	 50 40	original manifold
Water/metal ratio	11–12	7,3–9,6	
Water pressure range (bar)	120	180–215	manometer
Protective atmosphere (nitrogen)	$0,6m^3/h$	$0,65m^3/h$	flow meter

The initial chemical compositions (ingots for melting) as well as the final chemical compositions of prepared powders were determined and controlled by classical chemical analysis. No significant difference between initial and final compositions was observed. In **Table 1** the chemical compositions of selected Cu-Ag brazing alloys are presented.

The process parameters of water atomization are water pressure, tundish nozzle diameter, apex angle and diameter of water jets, superheating of the melt, etc. The main influent powder particle size parameter of these is water pressure[11] and water velocity, respectively. In the first stage of our experiments the process parameters of water atomization were established and optimized in order to get an optimal mean particle size and optimal particle size distribution. For brazing, first of all the powder fractions between 45 and 125 μm are commonly used. In **Table 2** the main controlled process parameters of water atomization during our experiments are presented.

All prepared powders were then sieved and their main morphological properties (particle shape, particle size distribution, mean particle diameter, apparent density and flowability) were determined. Besides the chemical composition of the alloy, these powder properties are the main applicability criteria of metal powders for brazing.

3 Results and discussion

All prepared powders were examined by optical and scanning electron microscopy (SEM). The micrographs show microstructures which strongly depend on the particle size and the cooling rate. **Fig. 4** and **5** show the typical microstructure of prepared powders. Some internal porosity of powder particles was noticed. **Fig. 6** shows the SEM micrograph of a water atomized Cu-2%Ni alloy-powder with nearly spherical particles. **Fig. 7** shows internal porosity of a water atomized Cu-Ag (L Ag15-P) based alloy-powder. Particularly Cu-Ag based alloys-powders show a large amount of powder particles with internal porosity.

The standard sieving analysis, as well as the Silas Alcatel laser granulometry of powders were also performed[4,5,12]. The powders have a regular and partially irregular particle shape. The mean particle diameter of prepared powders strongly depends on the water pressure used. The portion of irregularly shaped particles raises with increasing water pressure (increased collision between droplets of molten metal as well as partially solidified particles during atomization). The relatively high portion of regularly shaped particles confirms the sometimes forgotten fact[6] that water atomization can also produce nearly spherical powders, which is a condition for high apparent density and good flowability of powders.

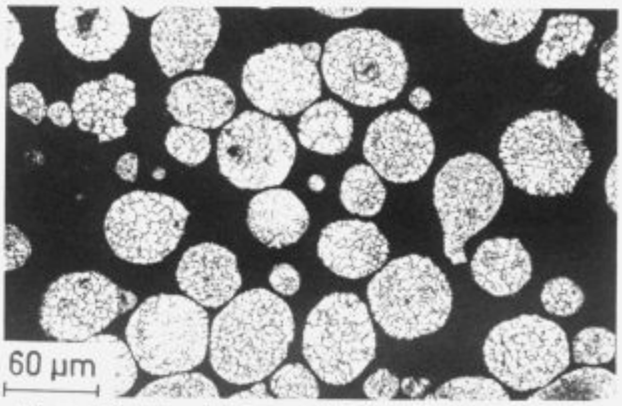

Figure 4: Cellular solidification of Cu-2%Ni alloy-powder particle.
Slika 4: Celična strjevalna mikrostruktura delca Cu-2%Ni prahu.

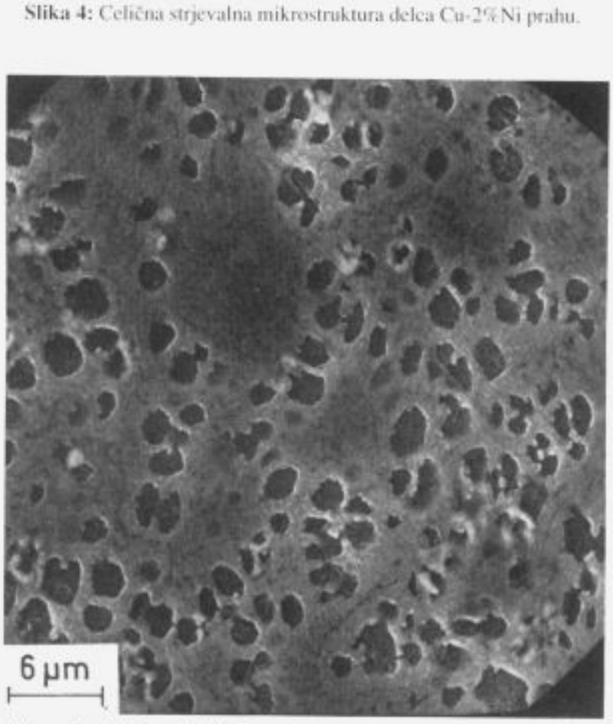

Figure 5: Cellular solidification of Cu-Ag (L Ag15-P) alloy-powder particle.
Slika 5: Celična strjevalna mikrostruktura delca Cu-Ag prahu (L Ag15-P).

The mean particle diameter of prepared Cu-2%Ni powders is approximately 50 μm at atomizing pressure 120 bars with a relatively regular particle shape and narrow particle size distribution (standard deviation σ = 2,2). The mean particle diameter of the prepared Cu-1,5%Cr powders is approximately 55 μm at atomizing pressure 150 bars with a higher amount of irregularly

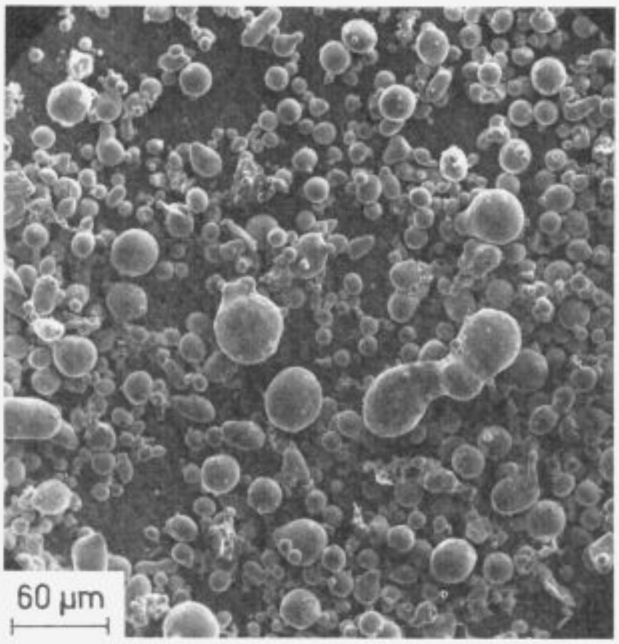

Figure 6: Particle shape of prepared Cu-2% Ni alloy-powder.

Slika 6: Oblika delcev pripravljenih Cu-2% Ni prahov.

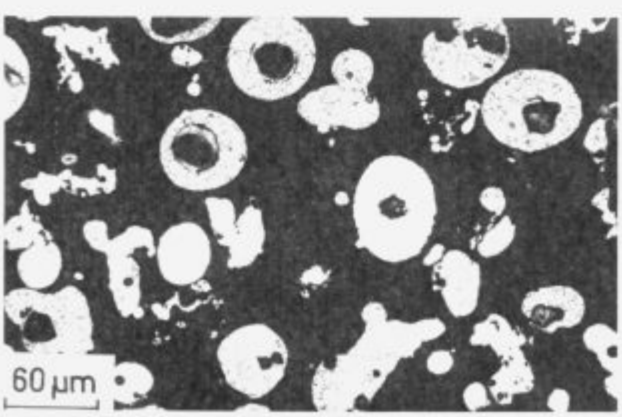

Figure 7: Internal porosity of nearly spherical Cu-Ag alloy-powder particles.

Slika 7: Notranja poroznost delcev Cu-Ag prahov.

shaped particles (high melting point Cr oxide formation) in comparison with Cu-Ni powders but a relatively narrow particle size distribution (σ = 2,1) is obtained. The preparation of Cu-Ag based powders was performed at considerably higher atomizing pressures (180 to 220 bars) in order to get a higher amount of particle size fractions below 125 µm. The mean particle diameter of the prepared Cu-Ag powders is approximately 70 µm at 180 bars and approximately 45 µm at the atomizing pressure 215 bars with a higher amount of irregularly shaped particles (increased collision) in comparison with Cu-Ni powders and a relatively narrow particle size distribution (σ = 2,25) is also obtained. Two informative atomizing experiments for the preparation of Au-Cu based alloys-powders (alloy with 58 mass % of Au - 14 carat gold and alloy with 75 mass % of Au - 18 carat gold) were also

Figure 8: SEM micrograph of Au-Cu water atomized powder with irregularly shaped (tear drop and ligamental) particles.

Slika 8: REM posnetek vodno atomiziranega prahu na osnovi Au-Cu z delci nepravilne (vlaknaste in kapljičaste) oblike.

performed. The mean particle diameter of the prepared Au-Cu powders is approximately 55 µm at atomizing pressure 140 bars with a relatively high amount of irregularly shaped particles (see **Fig. 8**).

Unfortunately our pilot water atomizer is too large (15 kg/batch) for the preparation of small quantities of the very expensive Au-Cu based alloys-powders which are normally used in practice. For these types of alloys-powders a small atomizer with the capacity of up to 0,5 kg/batch max. is suitable. We therefore decided to give up further experimental work until we purchase a small gas/water atomizer suitable for the preparation of these alloys-powders.

For other constant process parameters of atomization, the relationship between water pressure and the mean particle size shows a good, from the literature[4,5,6,11] well known exponential correlation ($D_{50} = k \cdot P_{H2O}^{n}$) for all prepared powders. The constants **k** and **n** depend on the alloy composition, the geometry of the atomizer and other process parameters of atomization. For the Cu-Ag based alloy (L Ag15-P type) the constants are estimated as **k** ≈ 8900 and **n** ≈ -0,96 for the tundish nozzle diameter 4,0 mm and the other process parameters of atomization given in **Table 2**. **Fig. 9** shows the experimental correlation: mean parti-

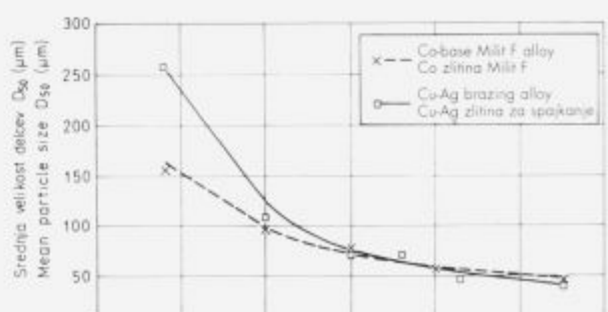

Figure 9: Mean particle size vs. water pressure for Cu-Ag brazing-alloy powder and Co-based welding-alloy powder.

Slika 9: Povprečna velikost delcev v odvisnosti od tlaka vode za prahove na osnovi Cu-Ag in prahove za navarjanje na osnovi Co.

cle size vs. water pressure for the Cu-Ag brazing alloy-powder and the Co-based welding-alloy powder. The influence of chemical composition is evident.

All prepared powders have the bimodal particle size distribution with two maxima at approximately 40 to 60 µm and approximately 100 to 180 µm virtually independent of the applied water pressure and other parameters of water atomization. This confirms the statement[13] that the disintegration of the molten metal stream during water atomization is carried out in two steps (the so-called primary and secondary disintegration). **Fig. 10** presents the sieving analysis of Cu-Al powders with noticeable bimodal particle size distribution and the influence of chemical composition. **Fig. 11** shows the bimodal particle size distribution of prepared Co-based water atomized powders determined by laser granulometry[9]. Bimodal particle size distribution with two maxima is clearly evident.

In the Wösthoffs apparatus the oxygen content of prepared powders was determined. The prepared water atomized powders have a relatively high oxygen content in spite of the fact that for pure metals (Cu, Ag, Ni and Au) Gibbs free entalphy for the reaction $Me+H_2O=MeO+H_2$ is positive[6,14] and therefore theoretical possibility for oxidation of pure metals with water steam is relatively low. Obviously, the alloying elements and impurities with higher affinity to oxygen (Cr, Mn, Cd, Zn, P, etc.) have an important influence on thin oxide film formation on the surface of powder particles as well as other factors which can increase oxygen content (discussed later in the article). **Fig. 12** shows that from the theoretical point of view, for some pure metals as well as alloys smaller oxygen contents in water atomized powders could be expected, and vice versa. In spite of that a rough estimation of oxygen content in water atomized powders is possible from this diagrammatic presentation. The diagram is based on literature[6] as well as our own experimental data.

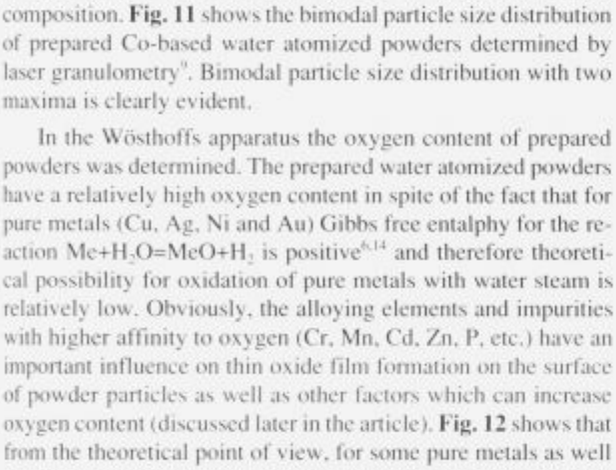

Figure 10: Sieving analysis histograms of Cu-Al water atomized powders.

Slika 10: Histogrami sejalnih analiz Cu-Al vodno atomiziranih prahov.

The plot oxygen content vs. particle size fractions exhibits, for most powders, the minimum oxygen content for particle fractions between 60 and 90 µm. The explanation of this phenomenon is in the combination of the effects of the particle size and the cooling rate (specific surface area - degree of oxidation) of particles during water atomization[1]. Exceptionally, powders with a high degree of particle shape irregularity exhibit a direct proportional increasing degree of oxidation with increased specific surface area of the powder[1]. **Fig. 13** shows the plot oxygen content vs. particle size fractions of different prepared powders with

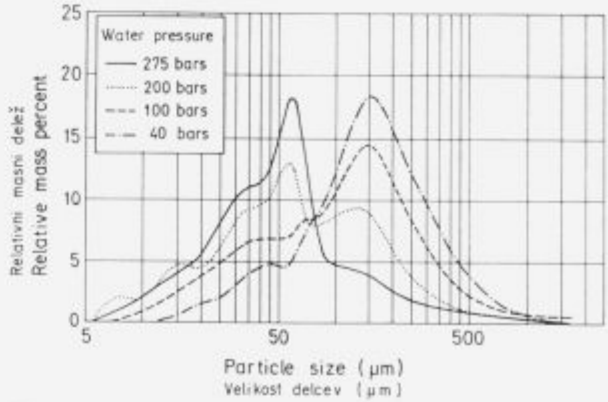

Figure 11: Bimodal particle size distribution of Co based welding alloy-powders performed by laser granulometry.

Slika 11: Dvogrba velikostna porazdelitev delcev prahov za navarjanje na osnovi Co dobljena z laserskim granulometrom.

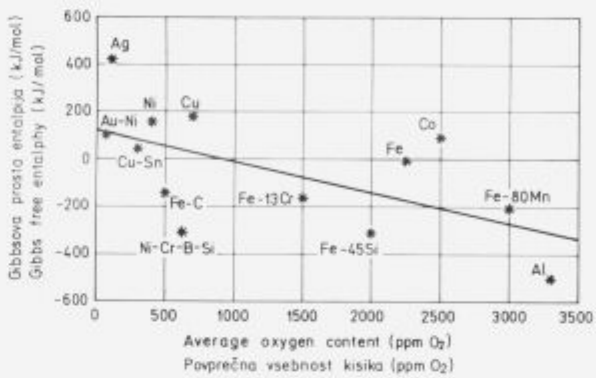

Figure 12: Gibbs free entalphy for the reaction $Me+H_2O=MeO+H_2$ vs. oxygen content for different water atomized alloys-powders.

Slika 12: Gibbsove proste entalpije za reakcije $Me+H_2O=MeO+H_2$ v odvisnosti od vsebnosti kisika za različne vodno atomizirane prahove–zlitine.

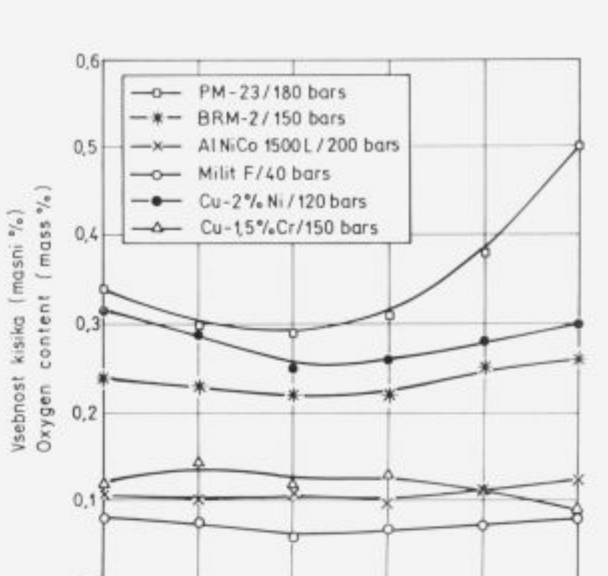

Figure 13: Oxygen content of prepared powders.

Slika 13: Vsebnost kisika v izdelanih prahovih.

the minimum oxygen content for particle size fractions between 60 and 90 μm.

The average oxygen content of prepared Cu-2%Ni powders is 0,23 mass % O_2, for Cu-1,5%Cr powders 0,15 mass % of O_2 and for Cu-Ag powders 0,15 mass % of O_2. Our opinion is that a considerable reduction of these values is possible by an additional optimization of water atomization (melting time reduction, superheating temperature as low as possible, inert gas blowing or protective slag used during melting, reduced water/powder particle interconnecting time, etc.). For example, the average oxygen content 0,075 mass % for Cu-Ag alloy-powder was already obtained during our experiments.

The relatively high oxygen content found in prepared Cu-based alloys-powders requires the consideration in what forms the oxygen can be found in powder particles. It is usually found in metal particles in the following forms: dissolved oxygen, surface oxides, surface adsorbed molecular oxygen and discrete oxides. The Ni and Cu-based powders can contain several thousand ppm of dissolved oxygen if not adequately deoxidised prior atomization[18]. The free energy for the reaction of Ni and Cu with water steam is positive, which suggests that the amount of surface oxides present in the Cu-Ni based powders is insignificant. The origin of discrete oxides is usually slag and refractory particles. The surface adsorbed molecular oxygen, which results from powder handling after the atomization, must not be neglected, either. In our future experiments, it would therefore be necessary to determine precisely the individual contribution of the above mentioned oxides to the total oxygen content of Cu-Ni powders.

The flowability and the apparent density of powders were determined with the Hall's apparatus[12]. The flowability raised with increased sphericity of powders. The fractions between 45 μm and 125 μm of all prepared powders have relatively good flowability but the particle size fraction below 45 μm has no flowability, except Cu-2%Ni and Cu-Ag powders prepared at the lowest (120 bars) water pressure. **Fig. 14** shows the correlation: flowability of prepared powders vs. particle size fractions. The powder fractions between 63 μm and 125 μm have the best flowability. The prepared Cu-2%Ni powders have the best flowability in comparison with other prepared powders. This is in accordance with the highest amount of regular (spherical) powder particles obtained for this type of alloy.

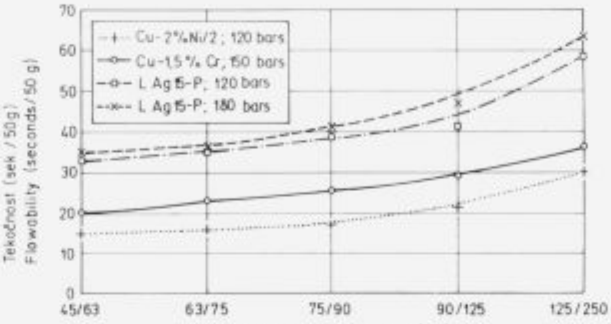

Figure 14: Flowability of prepared powders.
Slika 14: Tekočnost izdelanih prahov.

Fig. 15 shows the correlation: apparent density of prepared powders vs. particle size fractions. The highest apparent as well as tap densities are obtained for the finest fractions of powders. In accordance with high amounts of internal particle porosity of prepared L Ag15-P alloy-powders a relatively poor apparent as well as tap density of these powders is obtained.

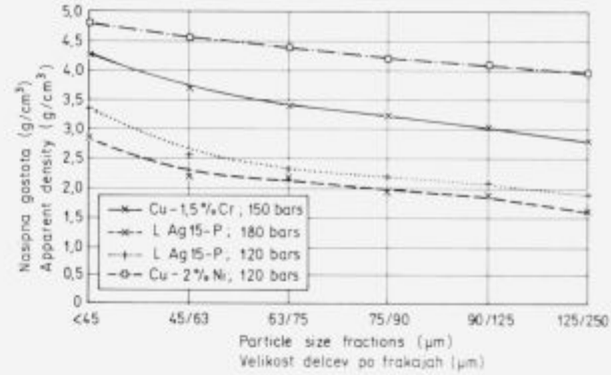

Figure 15: Apparent densities of prepared powders.
Slika 15: Nasipne gostote izdelanih prahov.

The determined morphological properties of prepared alloys-powders show that water atomized powders could be useful for different brazing applications, where relatively high oxygen content is not harmful (vacuum brazing, brazing in reductive atmosphere, brazing with alloy additions of deoxidizers or fluxes that produce light low melting point protective slags). Therefore we also made some brazing experiments[2,15,16]. Vacuum brazing of high speed steel (circular saw segments and paper knifes) on the structural steel base with a Ni-Cr based alloy (Nicrobraz Wall Colmonoy type 30 and LM filler metals) as well as with the prepared Cu based brazing alloy-powder gives high strength, non porous and other defect free, well diffusion bonded joints. **Fig. 16** shows a high temperature vacuum brazed high speed steel (circular saw segments) on the structural steel base with simultaneous heat treatment performed at IMT Ljubljana. **Fig. 17** shows the microstructure of a high temperature vacuum brazed tool steel/structural steel joint produced at IMT Ljubljana with a Cu-based brazing powder.

Vacuum brazing of sharp edged WC-Co particles on the steel plate also gives good results. **Fig. 18** shows vacuum brazing of sharp edged WC-Co particles on a steel plate (grinding wheel)

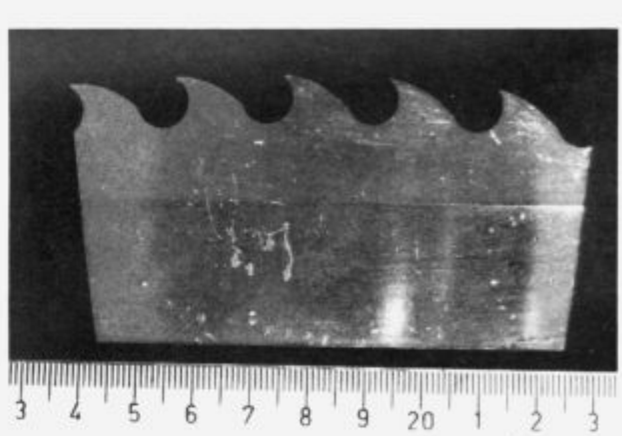

Figure 16: Vacuum brazing of high speed steel (circular saw segments) on the structural steel base.

Slika 16: Segment krožne žage iz orodnega jekla vakuumsko spajkan na osnovo iz konstrukcijskega jekla.

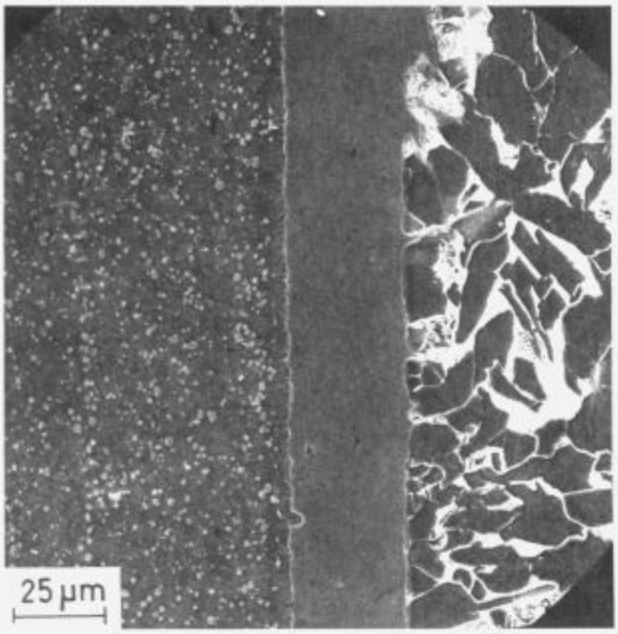

Figure 17: Microstructure of high temperature brazed tool steel/structural steel joint.

Slika 17: Mikrostruktura vakuumskega spoja orodnega in konstrukcijskega jekla.

with a prepared Cu-2%Ni brazing alloy-powder performed at IMT Ljubljana. Cu-Ag based alloys-powders also give good brazing results and the above mentioned brazing alloys are already produced on the level of small scale production for Slovenian electrical engineering industry (ISKRA Autoelektrika Nova Gorica, Metalflex Tolmin, etc.). **Fig. 19** shows industrial vacuum brazing of heating elements on the steel plate carrier performed at IMT Ljubljana. **Fig. 20** shows electrical components industrially brazed with water atomized Cu-Ag based alloys-powders.

4 Conclusions

The preparation of Cu based brazing alloys-powders by water atomization was investigated. The main morphological properties of prepared powders were then determined. The determined morphological properties of prepared alloys-powders, as well as practical brazing experiments show that water atomized powders could be successfully used in many brazing applications, especially where relatively high oxygen content in water atomized powders is not harmful. However, practical brazing experiments supported by metallographical and mechanical investigations of each individual brazing application are always necessary in order to confirm the usability of the selected water atomized Cu based alloy-powder.

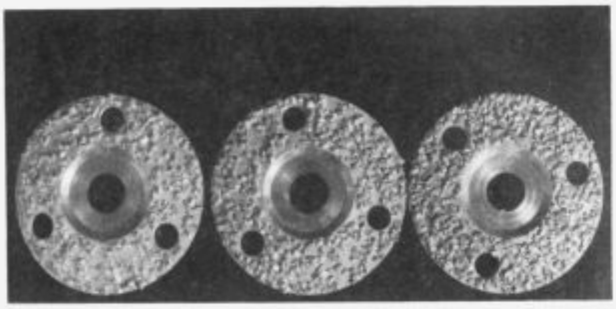

Figure 18: Vacuum brazing of WC-Co particles on the steel plate.

Slika 18: Vakuumsko spajkanje ostrorobih delcev karbidne trdine na jekleno osnovo.

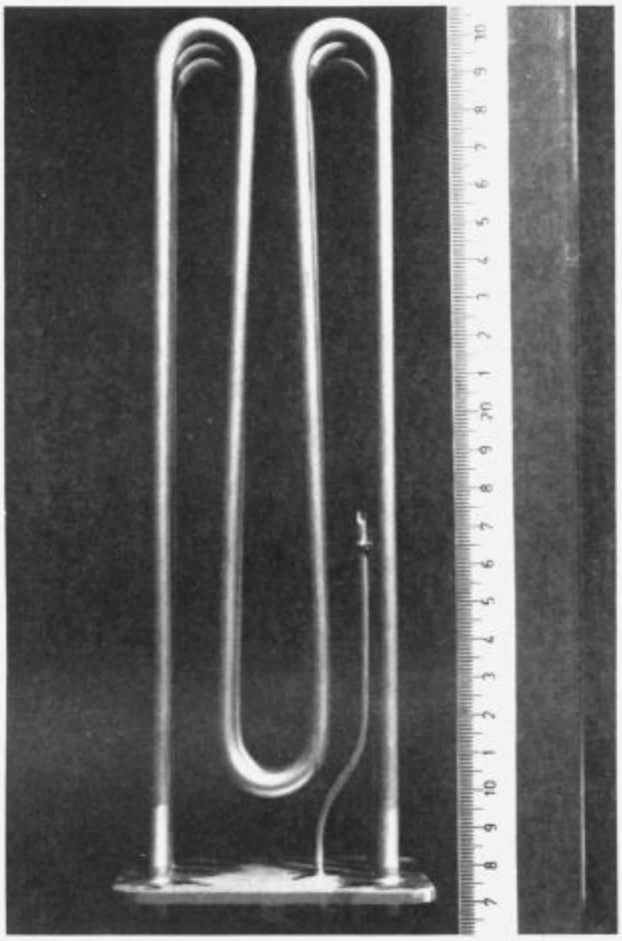

Figure 19: Vacuum brazing of boiler heating elements on the steel plate carrier.

Slika 19: Vakuumsko spajkanje grelnih elementov na jekleno nosilno osnovo.

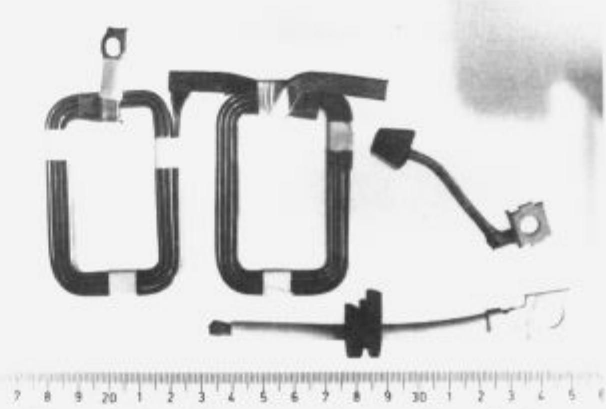

Figure 20: Electrical components for automotive applications industrially brazed with water atomized Cu-Ag based alloys-powders manufactured at IMT Ljubljana.

Slika 20: Električni sestavni deli za avtomobilsko industrijo spajkani z vodno atomiziranim Cu-Ag prahom izdelanem na IMT Ljubljana.

An important fact that has to be considered is the yield of usable metal powder (fraction +45 -125µm up to +45 -75µm) for brazing applications. Water and gas atomization give approximately the same but relatively poor powder yield (≈25÷35%). Centrifugal atomization gives a better yield (≈ 40%) but seems to present some practical difficulties in engineering and reliability[17]. Therefore, from this point of view, it can be concluded that water atomized powders for brazing are comparable with gas atomized powders.

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