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Development of Process Conditions for the Preparation of Copper Alloys Post-Production Chips for the Continuous Casting Process

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Abstract

The paper presents the results of research aimed at developing assumptions for the preparation of a charge in the form of fine scrap copper alloys (chips/shells) guaranteeing effective removal of impurities and obtaining a metal bath of the required metallurgical quality. The tests were conducted for tin-zinc-lead bronze of the CC499K grade. As part of the work, the characteristics of this type of waste available on the market were made in terms of quality and the possibility of their use for the production of both alloys and finished products, taking into account the elimination of harmful impurities that may ultimately affect the production process adversely.

The subject of the work was the selection of appropriate waste cleaning methods in the form of an oily shell in the CC499K (CuZn5Sn5Pb2) grade and its drying in terms of increasing the use of impure waste from machining as scrap for direct melting. The waste was assessed in relation to individual parametres. The research was carried out on 3 groups of waste, with varying degrees of moisture.

Keywords: Waste, Recycling, Free machining bronze, Moisture, Continuous casting

1. Introduction

Circular economy generates the need for full recycling of waste. At the same time, increasingly stringent environmental requirements significantly limit the possibility of harmful substances emission into the environment, including the atmosphere. One type of troublesome waste generated during metal machining are chips with a contaminated surface layer, additionally mixed with machining fluids. However, their full management is now a necessity [1, 2].

In addition, the growing requirements for manufactured products mean increasingly limited levels of permissible impurities. This problem is particularly acute for fittings that come into contact with water intended for consumption, and which, due to the need to use machining, must have good machinability and meet stringent requirements regarding the content of harmful impurities. An example of such a product is bronze CC499K (CuSn5Zn5Pb2), with a reduced lead content, widely used in many Western countries in the form of continuously cast products. The management of relatively large amounts of waste generated during machining of this alloy requires an increased share of very fine chips in the charge, which is often a major impediment caused by non-metallic impurities, mainly residues of machining liquids, the amount of which (due to the large surface area of the particles) is significant and difficult to remove. On the other hand, from the point of view of production economics, the use of this post-production waste is beneficial due to a lower price of this charge component [3].

Applied melting technologies usually strongly limit the permissible proportion of highly fragmented waste (the so-called "shell") and anticipate additional metallurgical operations using processing aids in the form of various types of fluxes. Surface contamination by volatile fractions is removed together with the waste gases from the furnaces, and the intensification of stirring the bath facilitates the melting process [4]. Removal of harmful oil contaminants by their prior burning in a rotary kiln, whereby flammable waste gases containing oil vapours can additionally be used to heat the kiln is also well-known and used in many plants. The resulting charge material is free of liquid contaminants but is characterised by an increased presence of oxides on the surface, which, when the chip surface is highly developed, constitutes a significant reduction in the yield of the melting process. In addition, this results in the emission into the atmosphere of harmful gases containing dioxins [5], formed at relatively low combustion temperatures, and results in the possibility of introducing additional sulphur contamination into the metal.

Another method, developed and used in the melting systems manufactured by Induga [6, 7], which is also the subject of patents EP 1055354 [8] and US 6240120 [9], is the continuous charging of a fine-particle charge onto a metal bath. In a special chamber built on top of the furnace, the chips are continuously fed from above by the feeding system, and when dispersed, falling from a relatively great height, they have the possibility of evaporating of the volatile fraction. As a result of its combustion, a reducing atmosphere is created over the surface of the metal, preventing metal oxidation, and while staying in the hot atmosphere caused by the burnt oil residue and on the surface of the liquid metal, the oil residues and emulsion from the surface of the chips are completely evaporated. As a rule, this method is largely consistent with the patent description PL74262 [10] with the difference that no protective flux coating is used. The advantage of this method of melting small scraps is the reduction of melting losses in a reducing atmosphere, the removal of potentially dangerous impurities present in oils (mainly sulphur), simplicity, relatively high efficiency, and good economy of the process. The disadvantage of this method is the incomplete burning of oil residues, the emission of harmful gases into the atmosphere, as well as a relatively high temperature of the exhaust gases, requiring their cooling before the treatment by filtering. A device of this type is also used in Poland [11] for melting lead brass machining waste, and the furnace is intended only and exclusively for melting of this type of charge.

The solution proposed in the publication eliminates most of these problems. The issue of contaminated post-machining waste is particularly acute in the case of chips which have not been subjected to effective centrifugation of machining fluids immediately after their formation, which during long storage on the surface of the particles have partly or completely dried oil residues. The aim of the presented research was avoiding the abovementioned disadvantages and inconveniences of using significant amounts of oily waste in the charge and reducing significantly the emission of harmful gases into the atmosphere. The technical challenge leading to this goal is to effectively remove oil residues from the surface of the chips before they are subjected to the melting process. The proposed solution is the removal of harmful oil residues before melting the charge, using a water bath with the use of chemical surfactants, mainly non-ionic surfactants, and using a specific process scheme, in a similar way as this solution is used for some products. So far, such a solution has not been used in relation to finely fragmented waste generated after machining. It should be noted that as waste, chips after machining, in accordance with the EU regulation [12], must meet the relevant criteria described in the relevant specifications and standards to be considered scrap approved for remelting, which ensures obtaining a good product.

2. Research material and methodology

The research conducted can be divided into three phases. In the first, performed on a laboratory scale, preliminary tests were carried out to select the type of a cleaning agent from commercially available products. In the second, also performed under laboratory conditions on the selected cleaning agent. Tests on the influence of the basic parameters on the efficiency of the cleaning process were carried out, and then the developed preliminary process scheme was verified under semi-technical conditions by replicating it on a larger scale of about 200 kg charge and running the process of continuous casting of bars on a laboratory machine. Subsequently, based on the results obtained, a verification of the process was carried out on the constructed pilot device, at industrial scale. The material used in the laboratory-scale tests was CC499K (CuSn5Zn5Pb2) bronze waste from three different sources (randomly marked S1, S2, S3), and produced after machining.

The waste assessment was carried out in accordance with the methodology specified in the PN-EN 12861:2018-07 Copper and copper alloys - Scrap standard, regarding the content of the magnetic fraction and moisture, with the measurement of moisture being determined in two stages as the evaporative fraction at 120°C and 350°C for information on the proportion of water and oily substances. The methodology for determining the melting yield in laboratory tests was also adopted according to the indications of this standard.

The measurements of waste grain size were conducted by performing a classic sieve analysis of dried chips, and then measurements of the specific surface area of individual chip size classes were carried out by means of the BET method using the Gemini apparatus by Micrometrics.

Standard equipment (a dryer, a water bath, a Digital Ultrasonic Cleaner PS-10A with a power of 70 W, a laboratory furnace, laboratory scales, etc.) was used for the tests on a laboratory scale. The effectiveness of the cleaning agents was assessed by measuring the share of oil residue after cleaning, and then relating it to the share of oil residue determined for the waste in the initial state after drying (roasting at 120°C) and presenting it as a percentage.

In semi-technical scale tests and during industrial scale verification, the degree of purification was assessed according to the indications of EN 12861. Chemical composition analyses were performed using a spectrometer (Thermo Scientific ARL 3460 OES). The tests of mechanical properties were carried out with the use of a testing machine (Instron 4505/5500K) according to PN-EN ISO 6892-1 standard, HBW hardness was measured using a hardness tester (Wolpert 2Rc) according to PN-EN ISO 6506 standard. The microscopic examinations were performed using the Olympus GX71 microscope.

3. Research on a laboratory scale

3.1. Waste characteristics

The basic parameters characterizing the waste used in the tests are presented in Table 1. This waste, stored for a long time, was characterized by a thin dried layer of oily substances on the surface, as evidenced by a very low share of moisture associated with the presence of water with a much higher share of oily substances. From the point of view of the purpose of the research, such an unusual state of the waste was beneficial, placing greater demands on the effectiveness of washing substances than for normally occurring in relation to waste not stored for a long period of time.

Table 1.
Basic properties of waste, wt %

Waste	Magnetic fraction share	Moisture evaporated at		Average specific surface
		120°C	350°C	
	%	%	%	m^2/kg
S1	0.002	0.046	1.378	83.894
S2	0.009	0.041	0.671	106.198
S3	0.045	0.039	1.238	77.984

The waste used was characterized by very low (incidental) contamination with steel chips, which eliminated the need for initial magnetic separation. Their common feature was a significantly developed specific surface of the chips with a dominant share of mainly fine fractions below 2 mm (Fig. 1), with the additional difficulty caused by the closed shape of the chips and their rough surface (Fig. 2).

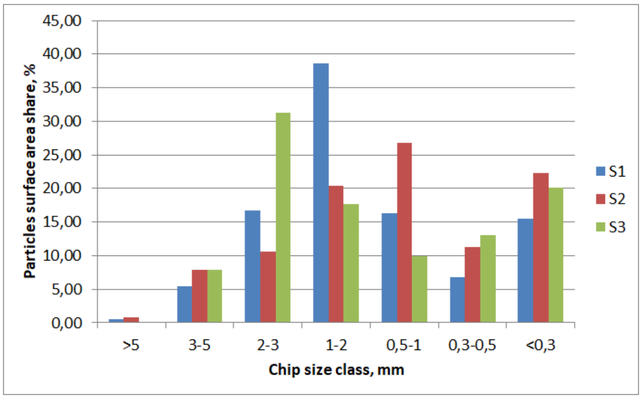

Fig. 1. Distribution of surface area of scrap chips particles

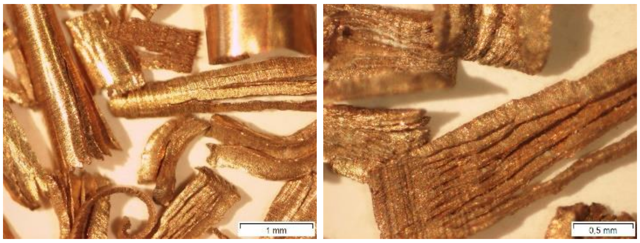

Fig. 2. An example image of the shape and surface development of the chips

3.2. Performance effectiveness evaluation of cleaning agents

The effectiveness evaluation scheme is presented in Table 2. Various commercially available products as well as hot water and steam were used as cleaning agents. The selection of parameters was guided by the manufacturers' recommendations. The results in the form of cleaning effectiveness, calculated as percentage scrap weight loss for individual waste and the average are presented in Figure 3.

Table 2.
Procedure for testing the efficiency of the treatment of various agents

Step	Description
1	Sampling - ca. 300 ml of scrap
2	Drying 120°C/12 h
3	Mass control, m_0
4	Cleaning solution application - 330 ml
5	Soaking in water bath: 65°C/2 h
6	Washing in ultrasonic cleaner: 60°C/30 min
7	Draining off the washing solution: 5 min.
8	Rinsing I with demineralized water: 60°C, ultrasonic cleaner
9	Draining off rinsing water, 5 min.
10	Rinsing II with demineralized water: 60°C, ultrasonic cleaner
11	Draining off rinsing water, 5 min .
12	Rinsing III with demineralized water: 60°C, ultrasonic cleaner
13	Draining off rinsing water, 5 min.
14	Drying: 120°C/16 h
15	Mass control, m_1
16	Roasting 350°C/5 h
17	Mass control, m_2

Remarks: Weighing was carried out with an accuracy of 0.001 g; repeatability of indications in the range of ± 0.002 g
Time of drying and roasting - removal of the moisture achieved by heating to temperature of 120 or 350°C until no more weight changes are detected - time 12/16 h for drying and 5 h for roasting was experimentally selected.

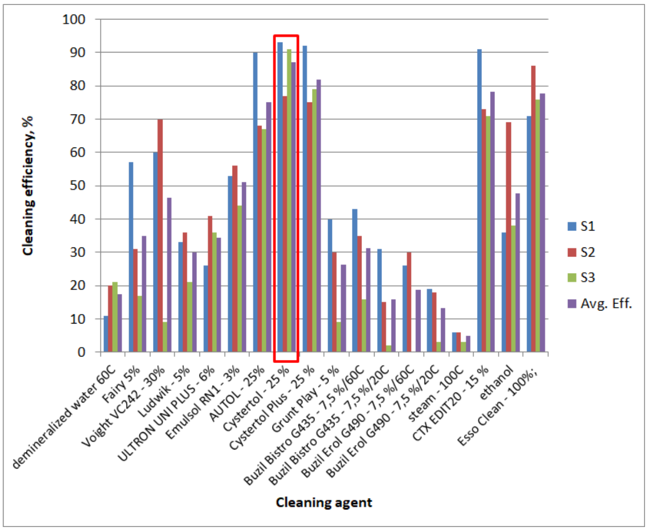

Fig. 3. Evaluation of the effectiveness of cleaning with various substances

The selection of the optimal cleaning agent considered also the data on the safety of use of the substances for humans and the environment, included in the safety data sheets of individual agents. Based on the obtained results, an agent manufactured under the name of Cystertol® was selected for further tests, being an alkaline degreasing and washing agent based on new generation, complex surfactants, intended for removing petroleum oils and grease. According to the manufacturer, the mixture of these agents effectively removes dirt, is environmentally friendly, and does not create smoke. It replaces turpentine, gasoline, diesel oil, diluter, and is used in the form of water solutions with a concentration of 5% to 50%, depending on the degree of surface contamination. It should be noted that the set of the tested cleaning agents also included other substances of a similar type, also characterized by good efficiency, which is visible in Fig. 3.

Based on the tests carried out under the same conditions as before, the effect of concentration and time (Fig. 4) as well as temperature were determined, and on this basis cleaning parameters of batches of waste for the melting and continuous casting tests were determined. Tests on the influence of temperature also showed its significant influence and the optimal value of approx. 60°C, in accordance with the manufacturer's instructions. In addition, it was found that the use of ultrasound in tests is less effective than mechanical stirring (Table 3).

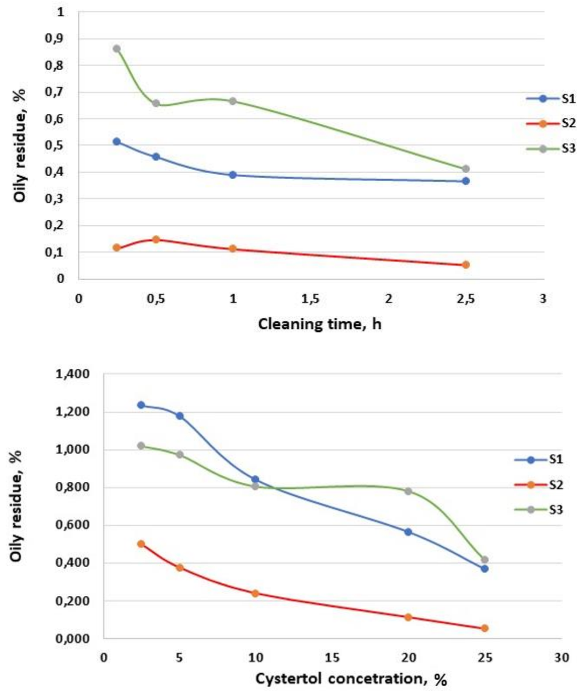

Fig. 4. Influence of concentration and cleaning time on the effectiveness of the dirt removal process (Cystertol®)

Table 3.
Influence of the mixing method on the effectiveness of oil fraction removal

Solution, concentration	Method	Temperature	Moisture[1)], %			Effectiveness, %		
		°C	S1	S2	S3	S1	S2	S3
Cystertol 25%	without mixing	40	0.492	0.131	0.685	**64**	**80**	**45**
	ultrasonic cleaner (70 W)	40	0.366	0.051	0.413	**73**	**92**	**67**
	mechanical mixing	40	0.033	-0.006	0.170	**98**	**100**	**86**

[1)] estimated error of residual oil measurement – 0.02%

3.3. Tests carried out on a semi-technical scale

Based on the results presented above, the conditions for cleaning tests on a larger scale were established. The conditions and results of waste treatment on a semi-technical scale are presented in Table 4. Due to the significant impact of the mechanical factor, waste treatment tests were performed in a rotating drum with a capacity of 140 l with the given parameters. They made it possible to clean approx. 200 kg of the waste from each of the tested batches. It is likely that the temperature of the cleaning solution higher than 40°C would increase the efficiency of the process even more, but it would require installation of additional heating of the drum and the overcomplication of the laboratory installation. In addition, the melting yield of the treated waste was assessed according to the procedure specified in PN-EN 12861:2018-07.

Table 4.
Conditions and results of waste treatment on a semi-technical scale (mean values for 4 cleaning tests)

Waste	Charge mass	Drum rotations cleaning solution	Cleaning temperature / time	Magnetic fraction, %	Residual water, %	Oily residue, %	Total moisture, %	Cleaning efficiency, %	Melting yield, %
S1	~50 kg	28 rev/min	40°C 15 min	0.002	0.07	0.43	0.50	69	99.08
S2		Cystertol 25%		0.005	0.05	0.10	0.15	85	99.44
S3		40 l		0.009	0.04	0.40	0.43	68	98.78

The chips obtained in the tests were used to make melts and evaluate the melt yield, and this material was then used as charge for continuous casting tests of ø15 mm and ø25 mm bars on the ConCastTech MINI SC laboratory casting line. The bar tests performed and the surface quality of the cast material were fully satisfactory and were the basis for running trials on a much larger industrial scale to verify the proposed process scheme.

3.4. Verification of the effectiveness of the process on the pilot line

The implementation of the research objective presented at the beginning of this article required the creation of technical possibilities adapted to the proposed method of waste treatment. These possibilities were provided by the built pilot line, the general scheme of which is shown in Fig. 5. The conditions for conducting tests verifying the proposed technology and basic parameters are presented in Table 5.

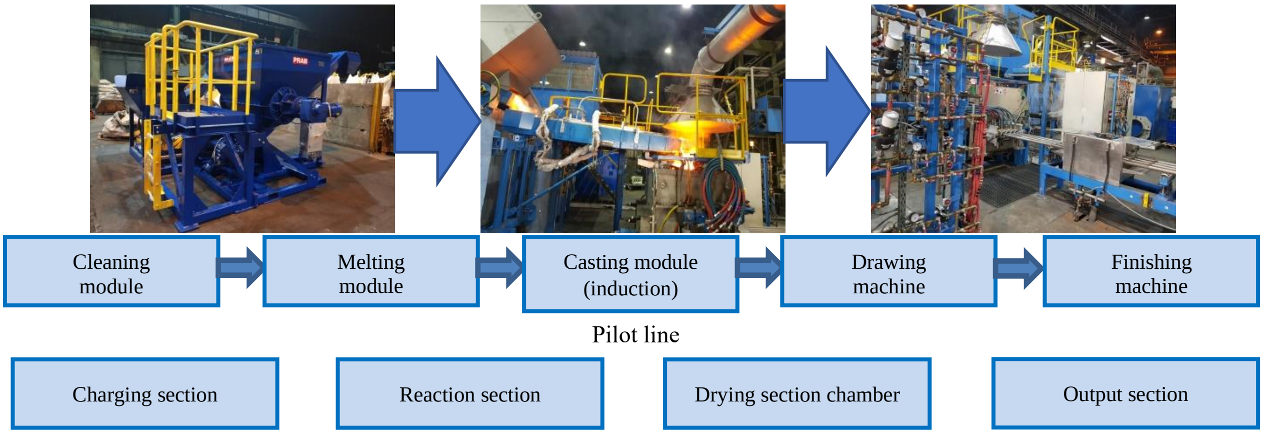

Fig. 5. General scheme of the pilot line and cleaning module

Table 5.
Test conditions and parameters on the pilot line

Material	CC499K
Charge	waste after machining
Mass of the charge	22 000 kg
Speed setting 1	Loading auger: 45Hz
Speed setting 2	Blower: 60Hz
Speed setting 3	Auger: 45Hz
Speed setting 4	Stirrer: 50Hz
Cleaning solution	Cystertol 13%
Stirrer working time	10 min.
Temperature	Ambient temperature
Cast product - charge	100% cleaned waste
Cast product 1	rod ø22 mm - 4 strands
Cast product 2	hollow rod ø57 × 12.5 mm - 2 strands

The results of the effectiveness of purification treatments carried out on the pilot line are shown in Fig. 6. The results of basic tests of cast products are shown in Table 6 (chemical composition of bars from individual melts) and in Fig. 7 and 8 (sample macro and microstructures), as well as in Fig. 9 (stability of mechanical properties). Table 7 summarizes the basic production indicators of the completed verification test.

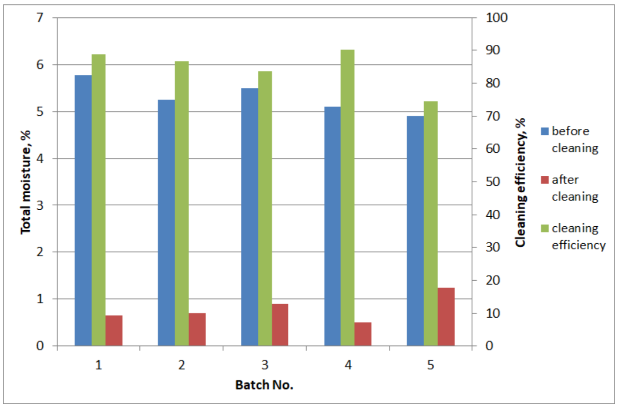

Fig. 6. The results of waste treatment effectiveness on the pilot line

Table 6.
Average bronze material CC499K composition with impurities and PN-EN 1982:2017 requirements

Value		Composition, mass fraction, %														
		Cu	Ni	Pb	Sn	Zn	Al	As	Bi	Cd	Cr	Fe	P	S	Sb	Si
required	min.	84.0	0.1	0.2	4.0	4.0	–	–	–	–	–	–	–		–	–
EN 1982	max.	88.0	0.60	3.0	6.0	6.0	0.01	0.02	0.02	0.02	0.02	0.3	0.04	0.04	0.1	0.01
mean		87.06	0.32	2.70	4.31	5.40	< 0.002	0.006	0.005	< 0.001	< 0.001	0.11	0.004	0.023	0.06	< 0.003
std. dev.		0.238	0.012	0.072	0.072	0.154	–	–	–	–	–	0.023	–	0.002	–	–
max		87.53	0.34	2.92	4.46	5.67	–	0.007	0.006	–	–	0.15	0.004	0.027	0.06	–
min		86.65	0.3	2.58	4.18	5.04	–	0.005	0.005	–	–	0.07	0.003	0.021	0.05	–

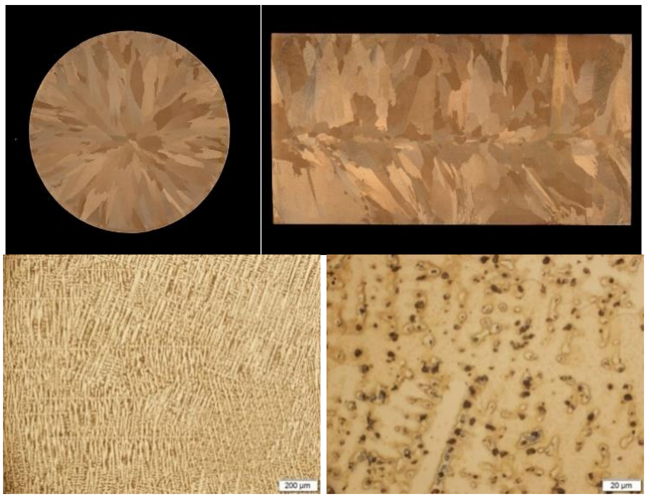

Fig. 7. Macro- and microstructure of cast rod ø22 mm

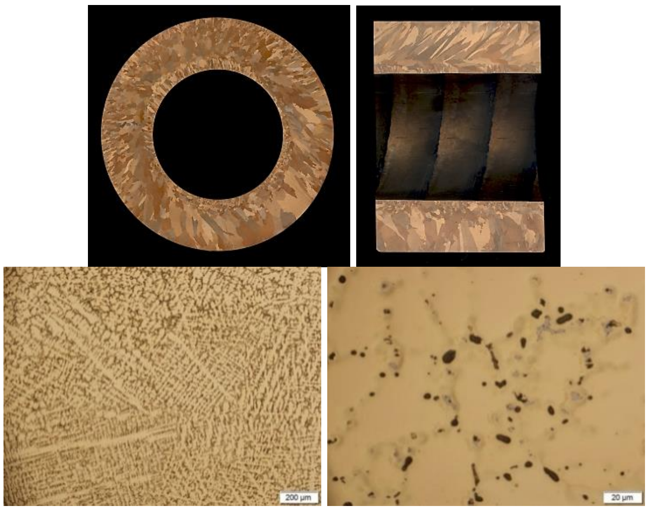

Fig. 8. Macro- and microstructure of cast hollow rod ø57 x 12.5 mm

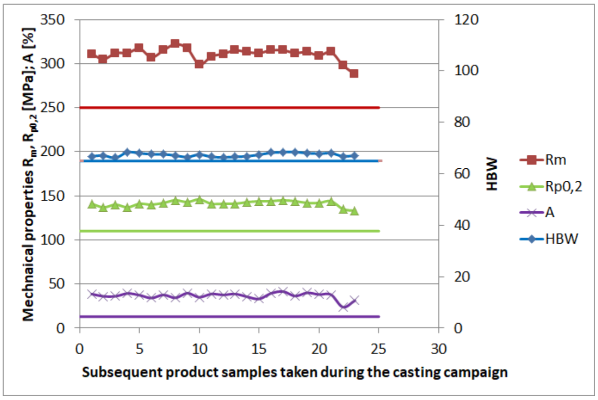

Fig. 9. Mechanical properties of cast rods. The required (PN-EN 1982) property levels are marked with straight lines in the figure

Table 7.
Technical and economic characteristics of the production of CC499K bronze from waste

Parameter	Value
Charge - cleaned chips	22025 kg
Cast semi-finished products	15832 kg (71.9%)
Technological waste[1)]	5636 kg (25.6%)
Dross	251 kg (1.14%)
Melting loss	306 kg (1.39%)
Metallurgical yield	97.5%
Energy consumption	1322 kWh/Mg

[1)] - leftovers from metal pouring processes during changeovers, crucible replacement stage of the holding furnace, and metal that was poured out from the melting furnace after the process was completed.

4. Summary and conclusions

The waste used in research conducted on a laboratory scale and then on a semi-technical scale was not a typical post-processing material, usually purchased on the scrap market. Due to the long period from creation to use in the research, they contained much less of the fraction that evaporates at 120°C (a defect), while the main part was the oil residue from the emulsion dried on the surface of the chips. Due to the nature of the material (free-cutting bronze), the chip particles were very finely fragmented, with a developed surface and a large share of very fine fractions, which was reflected in the results of surface area measurements (Table 1) - 80 ÷ 100 m^2/kg. Although this waste was not representative of the commonly found on the market, it was beneficial for the purposes of the conducted research, posing a great challenge for the tested cleaning agents and the methods of their application. The obtained results were used to determine the initial assumptions of the tests carried out on a semi-technical scale, and above all to select the type of degreasing agent. This made it possible to obtain (on a semi-technical scale) the reduction of oil residue at a level well below 1% and to achieve a satisfactory level of cleaning efficiency (Table 4). Also, preliminary tests of melting and continuous casting on a small scale on the ConCastTech MINI device gave fully satisfactory results both in terms of the process itself and the quality of the cast bars.

It should be emphasised that adding an additional interaction factor in the form of mechanical friction between the chip particles has a very significant effect on the cleaning result, which is particularly evident for waste with a layer of oil residue dried on the surface. Of course, many factors influence the cleaning efficiency, which is highly dependent on the type of waste being cleaned. For example, trials conducted under industrial conditions have shown that the optimum concentration of Cystertol can be lower (13%) than that used in previously conducted laboratory-scale trials (25%). Also, other cleaning agents of similar composition can be used.

The most important are the results of the verification of the cleaning process carried out on the pilot line (Fig. 5). The efficiency of this process is fully satisfactory, not only in terms of significant reduction of chip moisture. First of all, it is related to a very significant reduction of smoke during melting of chips and to obtaining cast products in the form of bars and sleeves with a chemical composition fully compliant with the requirements of the product standard (Table 6), as well as the required mechanical properties (Fig. 9). The quality of the surface of the products did not raise any objections and was similar to the products produced from lump charge. Similarly, metallographic examinations did not reveal the presence of internal casting defects, and the macro- and microstructure of the bronze was typical, with characteristic dendritic segregation depending on the cooling rate of the product (Fig. 6, 7).

Some of the techno-economic parameters of the process carried out, presented in Table 7, are the result of tests on a batch of 22 Mg of waste, while it should be noted that mastering the process fully requires further trials and the determination of optimum conditions also depending on the type of waste and the degree of contamination, which may improve it further.

The presented research allowed for the development of a new method of management of machining waste. It involves treating the waste with a water-based cleaning solution containing a mixture of non-ionic surfactants and other additives. Agents of this type, slightly different in ingredients, are available on the market under many trade names, and are used to clean equipment and technical installations from grease and oil residues, do not contain poisonous substances and are harmless to the environment. Exposing the waste to the solution, preferably at an elevated temperature to shorten the exposure time, with or without mechanical agitation, dissolves the residual oil present in the waste and forms a water-oil emulsion which is easily removed by centrifugation. The cleaned chips can be additionally rinsed in water followed by centrifugation, and the wastewater from the process can be reused or used to prepare a cleaning solution. This solution can be used many times, depending on the degree and type of waste contamination, and after its effectiveness decreases, it can be disposed of in a manner analogous to used rolling emulsions, emulsions for machining, liquids after cleaning installations and tanks in the petrochemical industry, bilge water, etc., in specialized companies. Technologies for the treatment of this type of wastewater have been known for a long time, and this method enables the recovery of oily substances and their disposal [13].

The main benefit is a significant reduction or elimination of the emission of harmful gaseous pollutants, improvement of the quality of the fine-grained charge for direct melting and the related improvement of efficiency indicators. Products obtained from the charge only in the form of cleaned chips are characterized by parameters identical to those from lump charge. Slightly increased process costs related to additional operations and waste management are compensated by more favourable financial conditions for the purchase of low-quality waste. The presented cleaning method is the subject of a patent application [14], and after gaining appropriate experimental experience on the pilot line, it can be the subject of wider dissemination in the non-ferrous metals industry.

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