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The Influence of Temperature on the Production of Antioxidant Tin-Phosphorus Alloy

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The restriction of lead content in alloys for the production of the solder based on the Directive of the European Parliament and of the Council of the European Union of 08.06.2011 which is also known as RoHS (Restriction of the use of certain Hazardous Substances in electrical and electronic equipment), had a very positive impact on the research of lead-free solder alloys as well as on the economic impact on the production of solders. It opened the door to issues relating to the mechanical properties of lead-free solders and the microhardness of formed joints, increasing their quality and efforts to reduce production costs. Besides the production efficiency increase, without the need for manual removal of so-called slagging, the moderation of oxide formation on the melt surface standing for the increase of the yield of the total amount of solder represents one of the many factors influencing the production of lead-free alloys for tin-based soldering. This work deals with the issues of material selection for the production of lead-free solders. Temperature affects the formation of different phases when there is the change in the concentration of the elements involved because it can be negative aspect for soldering. Therefore, it is necessary to have detailed knowledge on all the process which takes place during the temperature changes.

Keywords: Tin alloys, Antioxidant alloy, Chemical composition, Optical emission spectroscopy, Energy-dispersive X-ray spectroscopy

1 Introduction

The stability of tin-based phases above its melting point (232 °C) to atmospheric oxygen is very low and its change can be observed with the naked eye. The melt with a tin content of at least 99 % in the batch is silvery shiny at the beginning in the process and after only a few minutes, the surface turns with matte finish due to the formation of a layer of oxides on its surface. The oxide layer is mainly a problem in lead-free wave soldering, in the case where the lead-free solder alloy is applied on surface of PCB boards. The formation of oxides in this case causes a large loss of molten solder, increasing the total cost of production. Due to the fact that the density of lead-free tin-based solder in the liquid state is almost the same as oxides at the surface (density of molten tin is 6.980 kg/m^3, density of SnO_2 is 7.010 kg/m^3 and SnO is 6.450 kg/m^3), it is very difficult to separate slag from the melt [1]. Moderation or reduction of slag formation on the surface of the tin melt is possible either by the absence of oxygen access or through the addition of phosphorus to the batch [2,3]. Red phosphorus is very reactive and ignites at a lower temperature in comparison with the melting point of tin and therefore, it is necessary to add it in the form of a pre-alloy Sn-P to the melt. The production of this pre-alloy requires compliance with the temperature and amount of phosphorus, i.e. precise steps of loading into the furnace under strict safety measures. In addition to the need to use safety features, such as a protective suit, protective shield, gloves resistant to high temperatures, it is also necessary to ensure adequate ventilation. Based on the study of specialized literature, it was found that by adding the pre-alloy Sn-P to the melt of tin alloys, the number of oxides on its surface is significantly reduced [4,5,6].

2 Materials and methods

The design of the material and experimental methods is based on theoretical and practical knowledge. The experimental part consisted of two parts:

- Preparation of samples of SnP2 alloy under laboratory conditions,
- Analysis of dross amount formed on the solder bath surface.

2.1 Preparation of samples of SnP2 alloy under laboratory conditions

Tin with 99.98 % purity was used to create the SnP2 alloy (content of elements in the alloy - Sn 98 %, P 2 %) in the form of cut tin wire with 5 mm diameter and stabilized P4 red phosphorus in the form of dust for synthesis. The measured weight of tin before the formation of the alloy was 490 g and the measured weight of P4 phosphorus was 10 g. To create an intermediate layer between tin and phosphorus, due to their different melting point (Sn 232 °C, P4 586 °C), fine silica sand mixed with bentonite in a ratio of 1:1 was used. This technological process was chosen based on the experience of the previous experiment and the effort to create an Sn-P alloy. Without the formation of an intermediate layer separating the two components entering the melting process, phosphorus, due to its relatively low burning point (240 °C), would burn out before a chemical bond between it and tin would be formed before forming the desired Sn-P-based alloy. The batch consisted of the following layers (Fig. 1) to obtain optimum results:

- 10 g of P4 (A) red, phosphorus in form of dust, storage in vacuum desiccator,
- Interlayer – fine silica sand and bentonite in a ratio of 1:1 (B),
- Tin, with 99.98 % purity, in the form of cut wire with 5 mm diameter (C),
- Top layer — crushed coal (D),
- Sibral – temperature-resistant insulation material.

Fig. 1 *Preparation of Sn-P alloy batch*

In this order mentioned above, the individual parts of the batch were placed in a ceramic cup and placed in the muffle furnace. The initial temperature during the insertion of the crucible filled with all layers was 120 °C. The crucible was heated at a speed interval of 10 °C/min. The casting temperature of the samples was different: 600 °C for the first melt, 700 °C for the second, 750 °C for the third and 850 °C for the fourth melt. When the final temperature was reached, the crucible with the batch was held at this temperature for 20 minutes. The insulation material was removed, and the interlayer of fine sand and bentonite was broken with a graphite rod, and then floated to the surface of the melt and subsequently it was also removed. After that, the crucible was covered with insulating material again and placed back into the furnace for 20 minutes at the required casting temperature to allow a complete reaction between phosphorus and tin (Fig. 2). The measured temperatures and melting time of individual samples are available in Table 1, where temperature (T_A) and time (t_A) were recorded before the interlayer was taken, and temperature (T_B) and time (t_B) were recorded after the melt crucible was reinserted into the furnace.

Fig. 2 *Sn-P alloy after melting process*

After the specified time, the contents of the crucible were poured into a mould and the sample was removed after the alloy had cooled. After that, all samples were visually inspected from the aspect of the impurities on their surface. The cooling of the sample was followed by its preparation for measuring the chemical composition on a digital optical emission spectrometer Q4 TASMAN with a CCD detector (Fig. 3). This spectrometer is designed to analyse the chemical composition of metal solid samples. The spectrometer's analytical tripod consists of a pneumatic sample pressure that allows the sample to be firmly held at different heights, an analytical tripod with a top plate, and an analytical hole underneath which the electrode is placed. Argon gas passes through the analytical tripod. The sample on the spectrometer must be representative of the material to be analysed. In order for the spectrometer to measure with the highest possible accuracy and correctly, each sample must be properly prepared. The underside of the sample should be machined on both sides with a removal of material of at least 2 mm. No coolant or lubricating oils should be used for machining to avoid contamination of the sample.

Fig. 3 *Digital optical emission spectrometer Q4 TASMAN with a CCD detector*

Before the actual measurement, the area of each turned sample we decided to analyse was sanded on a grinder with new, uncontaminated sandpaper with fine grain thickness. The requirements for each sample measured were as follows:

- Electrical conductivity — the sample must be electrically conductive, without impurities on the surface.
- Strength — the sample must be solid and not porous, free from bubbles, cracks, and dirt.
- Homogeneity — the sample must be homogeneous throughout its volume and there should be no segregation of individual elements. The surface of the sample must be flat, clean, dry and unoxidized and the sample must be larger than the hole above the electrode in order to cover and seal it completely.
- Cleanliness – do not place the surface of the sample with the sanded side on the table to avoid contamination of the surface to be analysed.

Before each analysis, the electrode should be cleaned with a cleaning brush. The prepared sample was then placed on a plate of an analytical tripod so that it completely covers the gap above electrode. The sample must extend 1 mm beyond the edge of the electrode gap, or ideally as much as possible in the center of the sample where it is expected to be most homogeneous.

2.2 Analysis of dross amount formed on the solder bath surface

To verify the effects of the antioxidant alloy SnP2 on reducing the amount of oxides on the melt surface in the production of tin solder, we chose the production of solder type SAC305 with chemical composition, which is shown in Table 1. The content of the elements in the table is indicated with the maximum permissible content of individual elements according to the standard for this type of alloy J-STD-006C. This type of alloy is specifically mentioned as a substitute for lead alloy Sn63Pb37 (Sn 63 %, Pb 37 %). Performance characteristics of this alloy are very desirable, e.g. this alloy shows best-in-class yield, outperforms all SnCu-based materials, has low dross generation, excellent solderability due to fast wetting speed (in back-to-back tests 0.65 seconds compared to 1.00 second for SnCu-based materials) and delivers excellent performance across wide range of flux technologies. SAC305 complies with all requirements of RoHS Directive (Article 4.1 of the European Directive 2011/65/EU). Alloy specification for maximum Lead (Pb) Content = 0.07 %. As Ch. Yuanming at all. writes in the article Microstructure evolution and growth kinetics of intermetallic compound in SAC305/Ag and SAC305/Cu solder joints during solid-state aging [7], SAC305 is a lead-free alloy that has proven to be a suitable substitute for leaded solder precisely because of its good mechanical properties and relatively low melting point (melting point is from 217 °C to 219 °C), a solder pot temperature for wave soldering application is from 255 °C to 265 °C and for selective soldering recommended solder pot temperature is from 280 °C to 320 °C. Due to this temperatures, this type of alloy is also commonly used to stabilize and reduce the copper content in wave solder bath. The temperature difference in use is also confirmed by Diepstraten, Gerjan in his book Lead-free Soldering Process Development and Reliability, chapter Wave/Selective Soldering, the solder temperatures for selective soldering are slightly higher than for wave soldering (260 °C). However, they are much lower than lead-free hand soldering temperatures for rework wire (400 °C). Typically, the solder temperature is in between 280-300 °C. The smaller nozzles need more heat to get enough energy into the assembly and have complete hole fill. Some engineers do not want to have temperatures above 300 °C because of fillet/pad lifting or flux activation loss [8]. Also, the results of M. Yang study showed that a Cu6Sn5 scallop type layer with round grains having a strong texture was formed at the interface of the SAC solders/Cu systems. The addition of Ag decreased the IMC/liquid SAC solder interfacial energy and improved the wettability of the solder on the Cu [9].

Tab. 1 *Chemical composition in alloy SAC305 according to standard J-STD-006C*

Chemical element	Content in alloy [%]	Chemical element	Content in alloy [%]
Sn	balance	**As**	0.03 max
Ag	3.0 ± 0.2	**Ni**	0.01 max
Cu	0.5 ± 0.1	**Bi**	0.10 max
Pb	0.07 max	**Cd**	0.001 max
Sb	0.10 max	**Al**	0.001 max
Zn	0.001 max	**In**	0.05 max
Fe	0.02 max	-	-

Two solder baths (A and B) with the same material content were prepared and each of 300 kg batch of SAC305 consisted of the following parts:

- Tin (Sn), with 99.98 % purity, in the form of bars – 288.45 kg.
- Silver (Ag), with 99.98 % purity in the form of cut wire with 5 mm diameter – 9.0 kg.
- Copper (Cu), with 99.98 % purity in the form of thin wire with 0.44 mm* – 1.5 kg.

(*) This form of material was chosen for better melting of copper at lower temperature.

In one of the solder baths (B) was used deoxidation alloy SnP2 (Sn 98 %, P 2 %), in form of solid waffle shaped ingots (Fig. 4). Weighed amount that was added to the melting was 1,05 kg.

Fig. 4 *SnP2 alloy in waffle shaped ingots*

All the above materials were put into an electric industrial furnace with pot made of casted iron. The heat temperature was set on 300 °C. After an hour and a half, the temperature has been reduced to 255 °C degrees and tin-phosphorus deoxidation alloy was then added to the second melt (B) to bring the phosphorus level up to a range of 0.005 % - 0.007 %. Melted alloy was mixed. The first dross began to appear. Already at this stage, a difference was noticeable in the furnace with an alloy without added phosphorus and with added phosphorus. The phosphorus-free melt color on the surface showed yellow tones and was dull, while the surface color of the phosphorus-containing melt was silver-glossy. All the dross generated during this period was carefully skimmed from the surface of the melt and weighed every hour. The total melting time was 6 hours under stable conditions at a temperature 255 °C. After 6 hours, samples were cast from the melt without the addition of phosphorus (A) and from the melt with the addition of phosphorus (B) into the brass mold for samples that you can see in the Fig. 5.

Fig. 5 *Brass mold for samples of melted solder alloy SAC305*

It is also important to consider the negatives of adding Phosphorus-tin alloy to a tin bath in the manufacture of solder. K. Sweatman in the study with title The effects of phosphorus in lead-free solders states about effect of adding phosphorus into to solder bath on stainless steel. As well as being a powerful antioxidant for solder phosphorus can also break down the oxide film that gives stainless steel its resistance to wetting by molten solder [10]. Once the protective oxide film had been penetrated the molten solder wets the underlying Fe-Cr-Ni alloy, which then begins to dissolve in the solder [11]. The consequence is erosion of parts exposed to the molten solder and even perforation of the walls of the solder pot resulting in leakage of molten solder, which creates serious health and safety issues as well as damaging the machine. One solution to the problem of the machine erosion caused by the phosphorus added to control drossing was to make the solder pot of cast iron, which is more resistant to dissolution or to line the pot with titanium. Pumps and nozzles were made more resistant to wetting and erosion by applying ceramic coatings or a treatment that created a surface layer of metal nitrides that are much more resistant to wetting and dissolution [12]. However, this adds substantially to the cost of soldering equipment.

3 Results

3.1 Preparation of samples of SnP2 alloy under laboratory conditions

The chemical composition of the four samples was measured using the Q4 TASMAN digital optical emission spectrometer. The casting temperature of the samples was different (Tab. 2). The phosphorus and tin contents for individual samples are available in Tab. 3. The dependence of the casting temperature and phosphorus content of the alloy is graphically shown in Fig. 6.

Tab. 2 *Measured temperatures and time during melting of alloy Sn-P*

Sample No.	Weight of tin [g]	Weight of P4 [g]	T_A [°C]	t_A [min]	T_B [°C]	t_B [min]
1	490.03	10.09	599	21	602	20
2	490.12	9.98	709	22	712	21
3	490.08	10.03	746	21	750	21
4	490.02	10.01	849	21	851	21

Tab. 3 *Tin and phosphorus content of samples 1 to 4 casted at different temperatures*

Sample No.	Weight of tin [g]	Weight of P4 [g]	Starting temperature [°C]	Ø Casting temperature [°C]	Weight percent of Sn [%]	Weight percent of P [%]
1	490.03	10.09	123	600.5	99.93	0.07
2	490.12	9.98	121	710.5	99.42	0.58
3	490.08	10.03	120	748	99.10	0.90
4	490.02	10.01	120	850	97.96	2.04

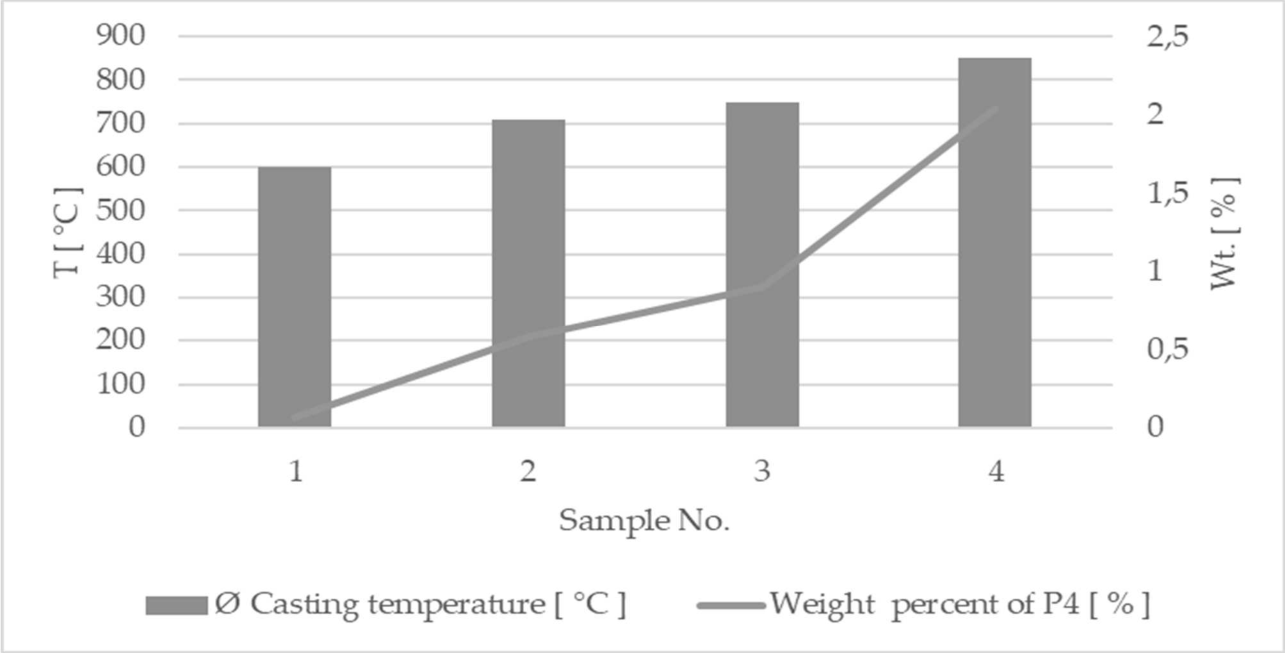
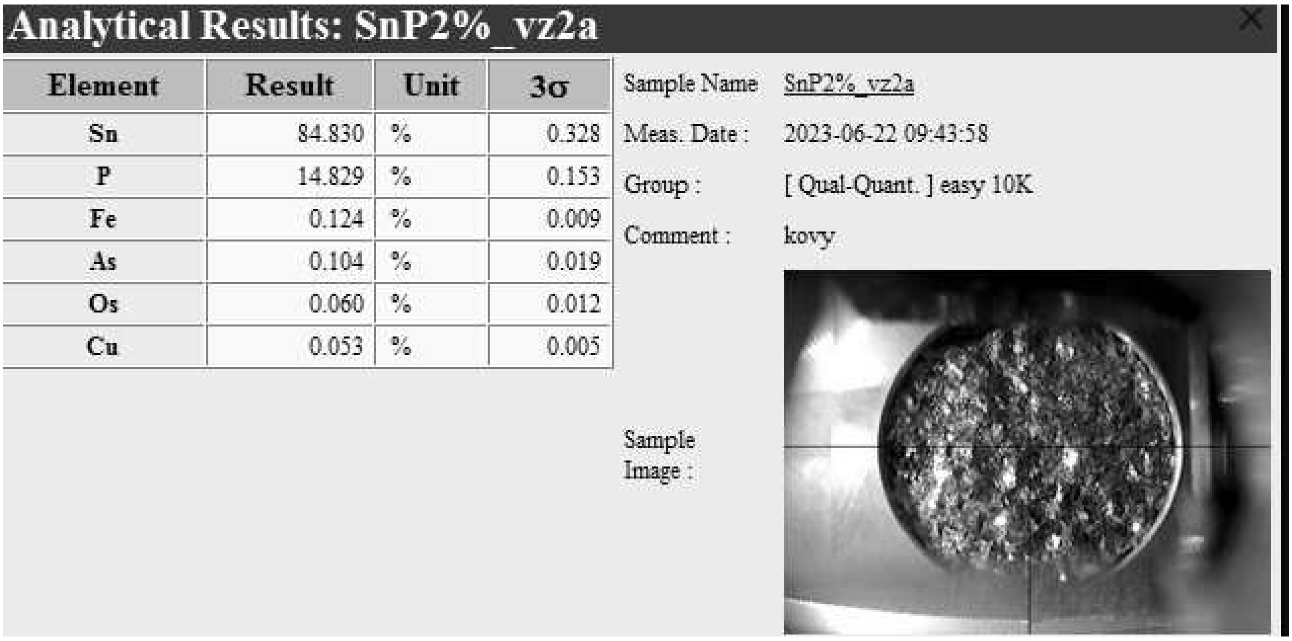


Fig. 6 *The dependence of the casting temperature on the percentage of phosphorus content*

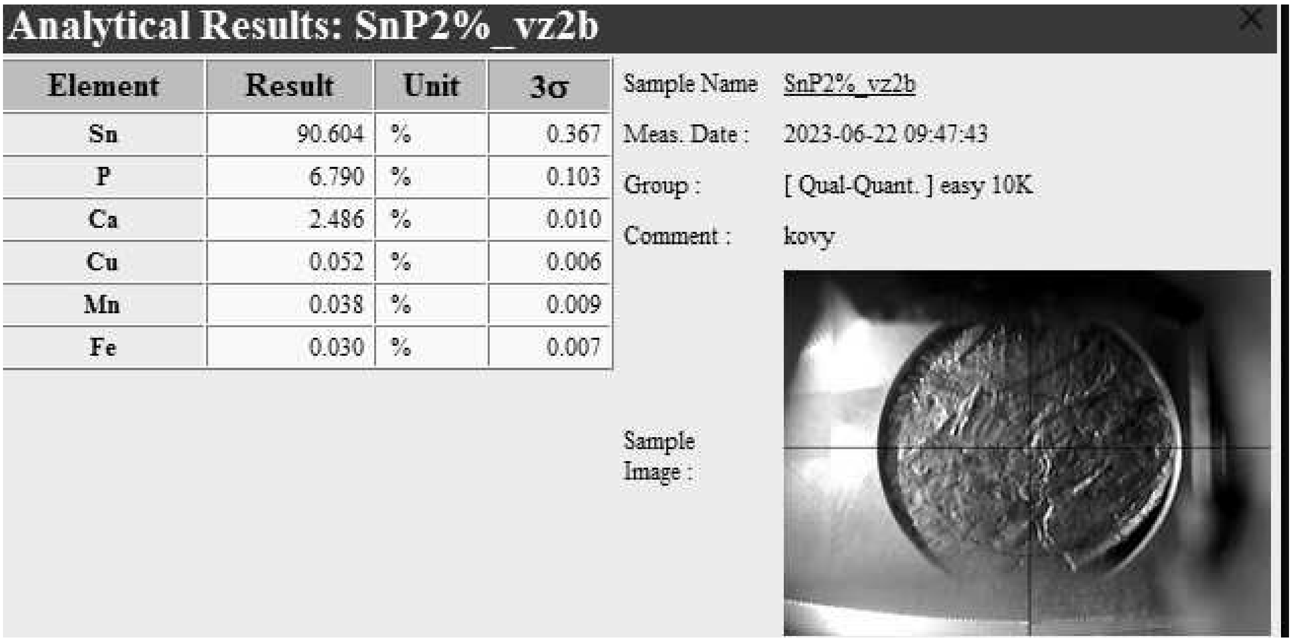
From the experiments and the results obtained and presented in the Tab. 2 and Tab. 3, it is clear that to form an Sn-P alloy with a phosphorus content of ≥ 2 %, the temperature higher than 800 °C is required for the reaction to take place the process completely. The observed surface of such a molten Sn-P alloy sample was rougher than that of the sample with a lower phosphorus content. A change in the colour and structure of samples surfaces could also be observed. This phenomenon in the casting of tin-phosphorus alloys is comparable to the specialized study which was introduced by AI-Ping Xian and Guo-Liang Gong: Oxidation Behaviour of Molten Tin Doped with Phosphorus [13]. The given study shows how the phosphorus content of the Sn-P alloy affects the oxidation of the casting surface. In the paper , there is the introduction of the fact that the oxidation resistance of a liquid alloy decreases with the amount of slag taken from its surface, which could be the result of unreacted phosphorus binding to slag forming oxides due to a lower melting point of the alloy. The light silvery surface of the sample remained constant only at a higher melting point. According to the occurrence of other elements in the alloy captured on the basis of the chemical composition measurement method using a digital optical emission spectrometer (Ca, Cu, Fe), the sample with the highest phosphorus content (Sample No. 4) was exposed to semi-quantitative measurement from the sample surface by energy disperse X-ray fluorescence spectrometry using the EDX-7000 Shimadzu measuring instrument. The results of this measurement show a more accurate content of the mass percentages of individual elements and indicate the purity of the alloy. The sample was measured from both sides and the measurement results are shown on Fig. 7 – Fig. 10.


Analytical Results: SnP2%_vz2a

Element	Result	Unit	3σ
Sn	84.830	%	0.328
P	14.829	%	0.153
Fe	0.124	%	0.009
As	0.104	%	0.019
Os	0.060	%	0.012
Cu	0.053	%	0.005

Sample Name SnP2%_vz2a
Meas. Date : 2023-06-22 09:43:58
Group : [Qual-Quant.] easy 10K
Comment : kovy
Sample Image :

Fig. 7 *Top of the sample No. 4 - chemical composition of the surface, measured by semi-quantitative method*


Analytical Results: SnP2%_vz2b

Element	Result	Unit	3σ
Sn	90.604	%	0.367
P	6.790	%	0.103
Ca	2.486	%	0.010
Cu	0.052	%	0.006
Mn	0.038	%	0.009
Fe	0.030	%	0.007

Sample Name SnP2%_vz2b
Meas. Date : 2023-06-22 09:47:43
Group : [Qual-Quant.] easy 10K
Comment : kovy
Sample Image :

Fig. 8 *Bottom of the sample No. 4 - chemical composition of the surface, measured by semi-quantitative method*

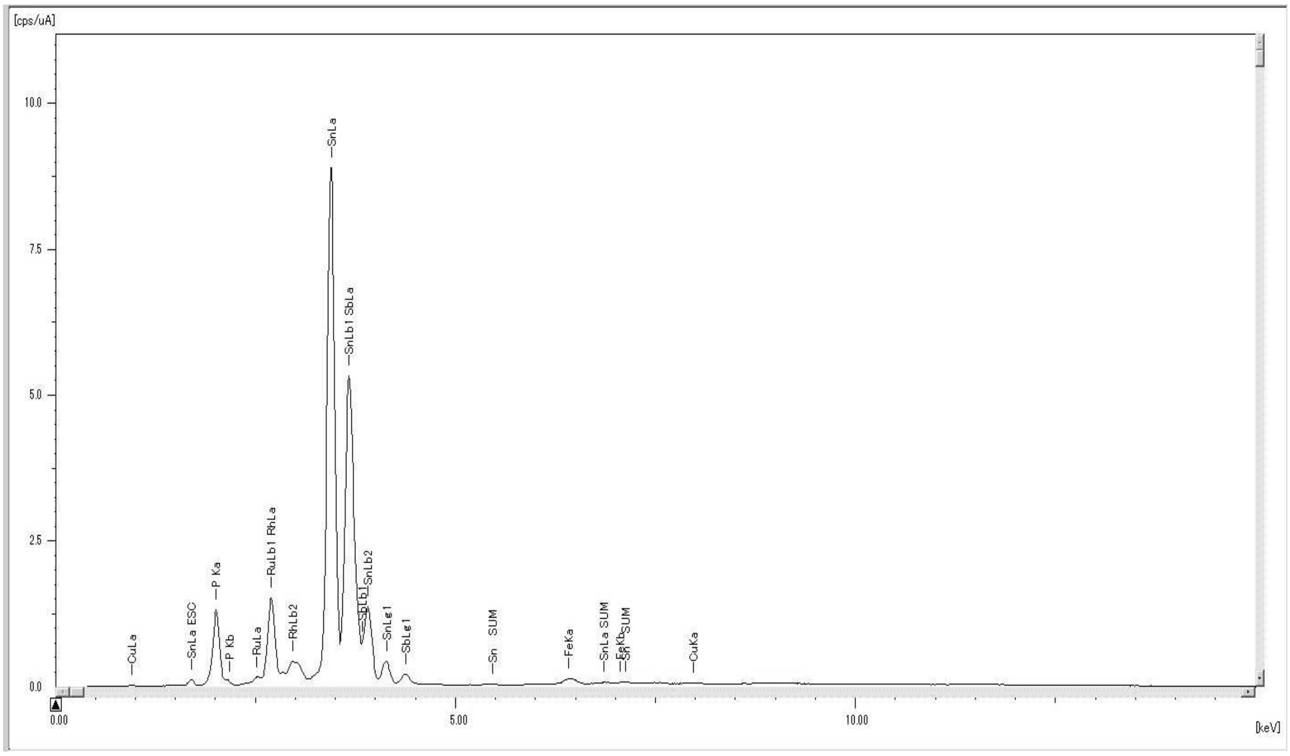


Fig. 9 *Top of the sample No. 4 - chemical composition of the surface, measured by semi-quantitative method*

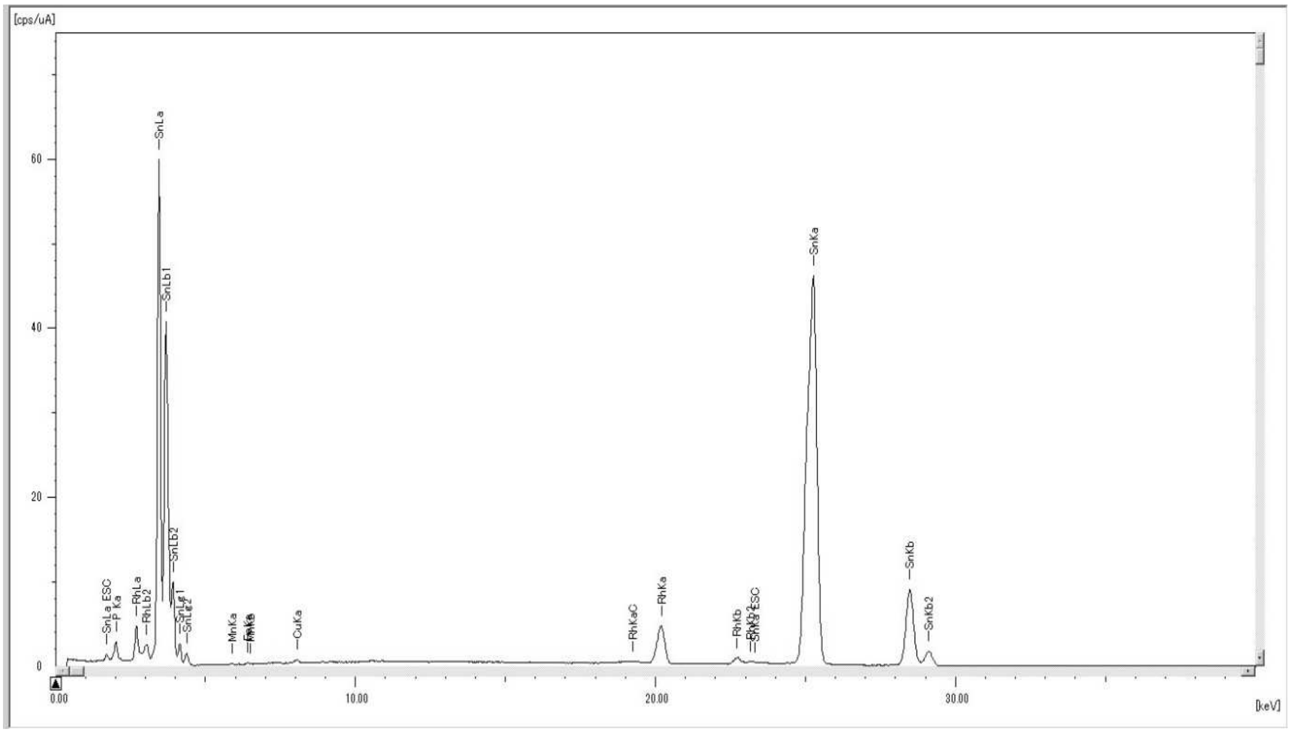
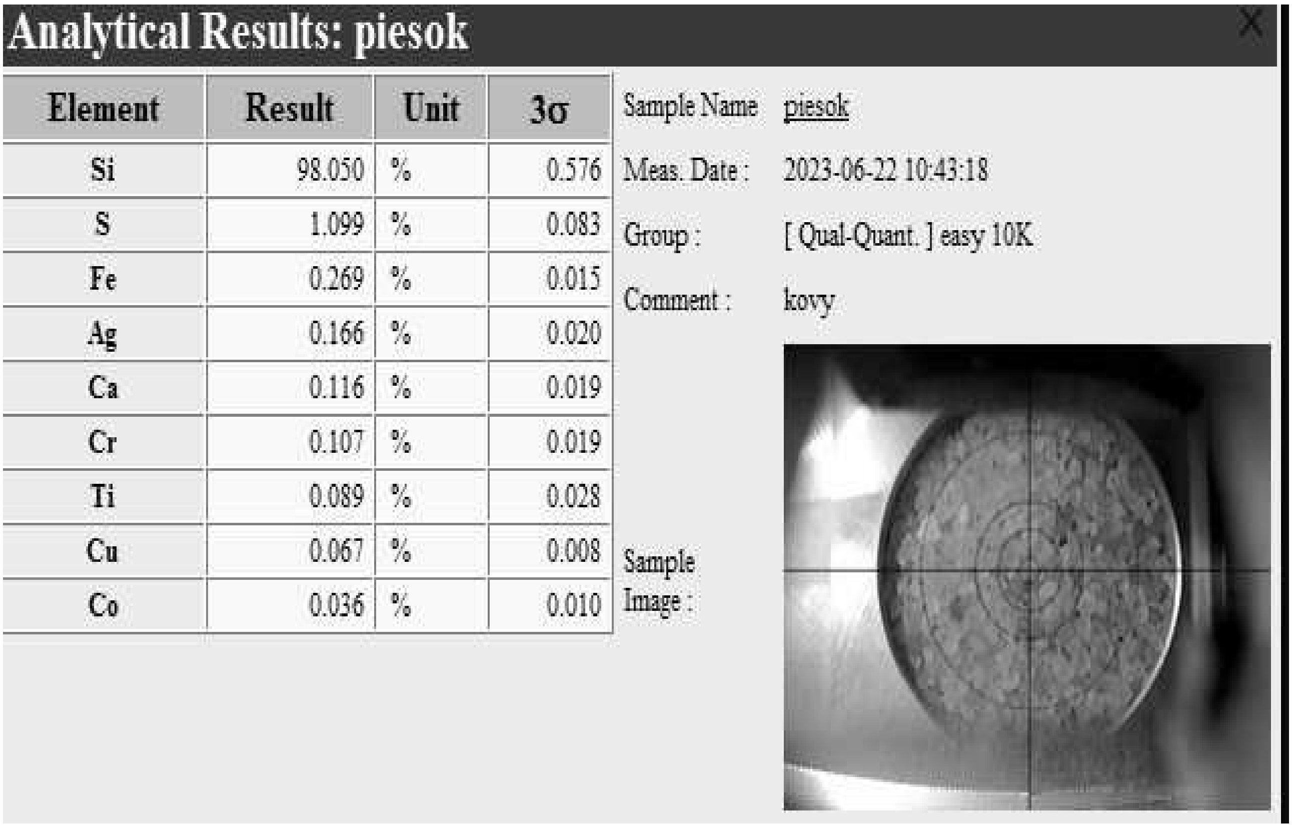


Fig. 10 *Bottom of the sample No. 4 - chemical composition of the surface, measured by semi-quantitative method*

It was the presence of calcium in group of the elements found in the Sn-P alloy that manifested itself in a higher percentage, which led to the measurement of the chemical composition of sand, and it served as an intermediate layer between the individual elements during the melting of the alloy and was considered as a source of calcium in the resulting sample [14,15]. The sand was also subjected to semi-quantitative analysis by energy disperse X-ray fluorescence spectrometry (Fig. 11). The measurement result was not satisfactory because the presence of calcium in the sand was negligible and probably it was not contaminant of the sample (Sample No.4).



Analytical Results: piesok

Element	Result	Unit	3σ
Si	98.050	%	0.576
S	1.099	%	0.083
Fe	0.269	%	0.015
Ag	0.166	%	0.020
Ca	0.116	%	0.019
Cr	0.107	%	0.019
Ti	0.089	%	0.028
Cu	0.067	%	0.008
Co	0.036	%	0.010

Sample Name piesok
Meas. Date : 2023-06-22 10:43:18
Group : [Qual-Quant.] easy 10K
Comment : kovy
Sample Image :

Fig. 11 *Sand used as an interlayer in alloy melting - chemical composition of the surface, measured by semi-quantitative method*

3.2 Analysis of dross amount formed on the solder bath surface

Two different samples were cast. Already during the cooling of the samples in the brass mold, there was a difference in the amount of oxides on their surface which can be observed with the naked eye (Fig. 13). The surface of the sample from the melt without the addition of phosphorus (A) had a significantly larger amount of oxides on its surface than the surface of the samples from the melt with most of the phosphorus (A). This significant difference is visible also in the Fig. 12. This visual difference refers to the ability of dross to form on the surface of both melts.

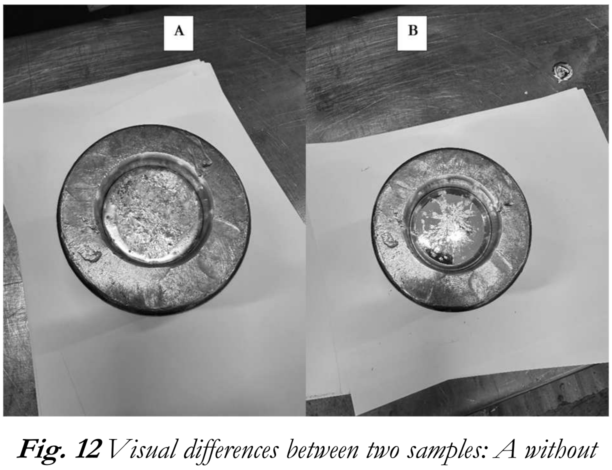


Fig. 12 *Visual differences between two samples: A without addition of phosphorus in the solder bath, B with addition of phosphorus in the solder bath*

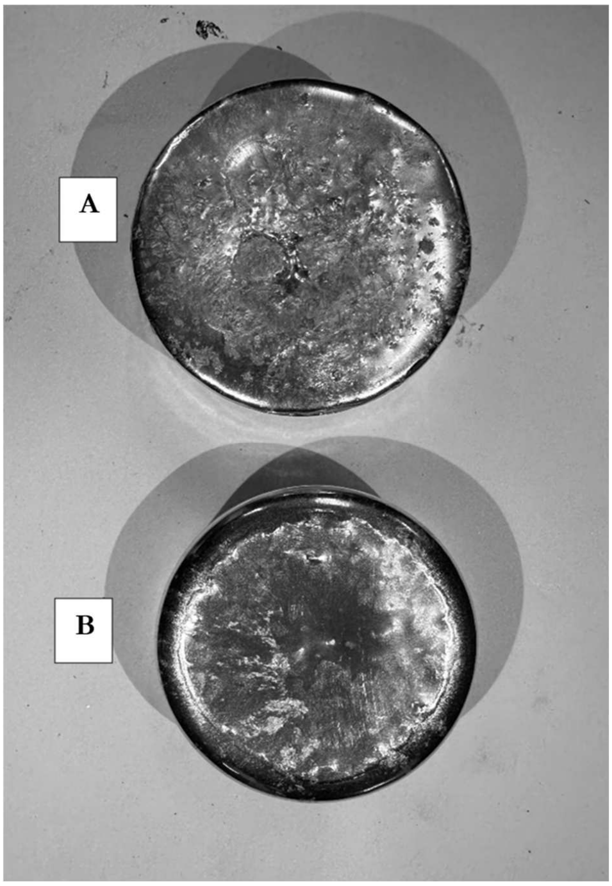


Fig. 13 *Visual differences between two samples – yellow tones of oxides on sample A surface, shine and glossy surface with significant less amount of oxides on sample B surface*

The cooling of the sample was followed by its preparation for measuring the chemical composition on a digital optical emission spectrometer Q4 TASMAN with a CCD detector. Tolerances for the content range of individual elements have been respected according to standard J-STD-006C. Chemical composition of sample A can be seen in Tab. 4. In Tab. 5, where the measured chemical composition values for sample B are shown, the phosphorus content is 0.003 %. At the beginning of melting, its content was higher (represented by 0.007 % in the total batch volume). If we consider the properties of phosphorus at higher temperatures [16, 17], it can be assumed that under the influence of temperature and melting time a certain amount burned out.

Tab. 4 *Chemical composition of sample A - without addition of phosphorus in the solder bath*

Chemical element	**Content in alloy** [%]	**Chemical element**	**Content in alloy** [%]
Sn	96.39	**As**	0.01
Ag	2.9	**Ni**	0.001
Cu	0.6	**Bi**	0.02
Pb	0.02	**Cd**	0.000
Sb	0.05	**Al**	0.001
Zn	0.001	**In**	0.02
Fe	0.01	**P**	**0.000**

Tab. 5 *Chemical composition of sample B - with addition of phosphorus in the solder bath*

Chemical element	**Content in alloy** [%]	**Chemical element**	**Content in alloy** [%]
Sn	96,42	**As**	0.002
Ag	3.0	**Ni**	0.005
Cu	0.4	**Bi**	0.004
Pb	0.02	**Cd**	0.000
Sb	0.1	**Al**	0.001
Zn	0.001	**In**	0.02
Fe	0.02	**P**	**0.003**

The dross taken from each melt every hour was weighed and recorded. The weighing results of dross from two solder baths SAC305 (A) and SAC305 with additon of phosphorus (B) are shown in the Fig. 14. The quantity of dross resulting from solder bath B is 35 % lower than that resulting from solder bath A.

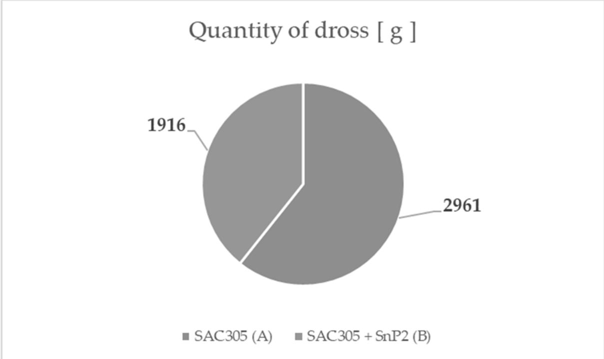


Fig. 14 *Quantity of dross from melts A and B*

4 Conclusion

In conclusion, based on the obtained relevant results, the melting process of Sn-P alloys must be performed in a clean and inert environment, due to the activity of con-stituent elements that can create other undesirable chemical reactions and connec-tions. The temperature and melting time must be maintained and monitored continuously. During the melting, the temperature increased, and while the holding time of approx. 21 minutes at a constant temperature seemed sufficient to carry out a chemical reaction between tin and phosphorus, the difference in the percentage content of phosphorus in the cast sample was influenced by the temperature. However, a tem-perature of 850 °C proved to be the most suitable for casting SnP2 pre-alloy, given that at this temperature the highest percentage of red phosphorus was contained in tin. In the process of melting this pre-alloy, it should not drop below this temperature before casting, if we want to achieve the highest possible phosphorus content during melting.

The dross taken from each melt every hour was weighed and recorded. The weighing results of dross from two solder baths SAC305 (A) and SAC305 with addition of phosphorus (B) are shown in the Figure 14. The quantity of dross resulting from sol-der bath B is 35% lower than that resulting from solder bath A. The formation of oxidic phases is undesirable for the further use of these alloys in the form of solders. The ad-dition of a tin-phosphorus deoxidation pre-alloy is one option to reduce the formation of dross on the surface of the melt during the production of solder. It is important to mention that the advantage of phosphorus in solder is not only the reduction in the weight of the smelting by-product (dross) in the manufacture of solder. The Sn-P pre-alloy has a high use in wave soldering, where it is gradually added to the melt in small quantities, significantly reducing the amount of oxides on the surface of the melt in the process of soldering PCB boards ctivity of constituent elements that can create other undesirable chemical reactions and connections. An element that behaves similarly in solder as phosphorus is germanium [18]. Important factor in melt production with the amount of returning material is to know to set its optimal amount and the way of melt treatment to achieve the same results in casting quality in comparison to the casting production from clean materials [19]. The temperature and melting time must be maintained and monitored continuously. The formation of oxidic phases is undesirable for the further use of these alloys in the form of solders. The addition of a tin-phosphorus deoxidation pre-alloy is one option to reduce the formation of dross on the surface of the melt during the production of solder.

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