THE NON-DESTRUCTIVE PREDICTION OF THE ALUMINIUM CONTENT IN PRESSED SKULLS OF ALUMINIUM DROSS

Scientific paper UDC: 669.71.054.9

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Abstract

During production of primary and secondary aluminium, various amounts of aluminium dross, a mixture consisting of molten aluminium metal and different oxide compounds, is skimmed per tonne of molten metal. In order to preserve the maximum aluminium content in hot dross for further extraction, it is necessary to cool the dross (e.g. by pressing) immediately after skimming. During pressing, the skimmed dross is transformed into so-called pressed skulls, convenient for storage, transport or further inhouse processing. Pressed skulls, which represent a valuable source of aluminium, are generally valued on a free-metal recovery basis. Therefore, it is important and useful to develop a method of fast and cost-effective non-destructive measurement of the free aluminium content in pressed skulls, independent of the technology of pressed skulls recycling.

Following the theoretical considerations presented in this work, a practical industrial methodology was developed for non-destructive prediction of the amount of free aluminium in pressed skulls, w_{Al} , based on non-destructive measurement of the density, o, of the pressed skulls.

Key words: Pressed skulls, aluminium dross, non-destructive prediction

Introduction

Aluminium dross appears as an unavoidable by-product of aluminium melting. The mechanism of dross formation is rather complex, consisting of surface oxidation of the melt, crushing of the oxide skin by bath movement, sinking and floating of oxide particles, conglomeration of oxide particles, filling up of interspaces by molten aluminium, internal oxidation of aluminium droplets dispersed in the dross, skimming of dross from the melt bath surface and metallic aluminium drip off following oxidation of solids during dross cooling. Although the formation of aluminium dross causes aluminium losses (because of oxidation of the melt and of filling up dross interspaces by molten aluminium), dross formation and complete skimming are also useful, enabling cleaning of the melt. Regarding chemical composition, aluminium dross is a

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mixture of free metallic aluminium (10-90%), non-metallic constituents such as oxides (Al₂O₃, Al₂O₃ MgO, Al₂O₃ SiO₂, Al₂O₃ FeO etc.), nitrides (AlN), chlorides (AlCl₃, KCl, NaCl), fluorides (CaF₂, NaF, AlF₃, Na₃AlF₆ etc.), carbides (Al₄C₃), sulphides (Al₂S₃), phosphides (AlP), dirt and impurities. The amount of dross formed/skimmed depends on different factors like the type and quality of input material (primary aluminium and/or aluminium scrap), operating conditions and type of technology, as well as the furnace applied. During the process of primary aluminium production, more than 40 kg of Al dross is generated per ton of primary aluminium and about 200 kg of Al dross is generated per ton of secondary aluminium [1]. Based on the world production of primary and secondary aluminium in 2011, a quantity of about 3 million tons/year (tpy) of aluminium dross skimmed worldwide may be estimated, representing a valuable source of more than 1 million tpy of aluminium (approx. 2% of world aluminium production). However, to preserve as much aluminium content in skimmed dross as possible for further extraction, it is most important to prevent, or at least significantly reduce, its further oxidation.

In order to minimise oxidation of valuable molten aluminium with which skimmed dross is impregnated, the hot dross, Fig.1, should be rapidly cooled – immediately after skimming, impeding at the same time the contact of molten aluminium in dross with air. One way to do this is to press the skimmed hot dross in a press, Fig. 2, which is popular for cooling hot dross and obtaining the maximum inhouse recovery of molten aluminium [2]. During that process, the skimmed dross is transformed into so-called pressed skulls, Figs. 3-5, with characteristic geometry convenient for storage, transport or further in-house processing. Part of the skulls is recycled internally, inside the home cast-house; the rest is dedicated for external recycling. Apart from internal or external routes of recycling, pressed skulls are generally valued on a free-metal recovery basis - as the percentage of aluminium recovered in a rotary furnace.



Fig. 1. As-skimmed aluminium dross before cooling by pressing.

The main problem with valuation of pressed skulls on a free-aluminium recovery basis is in their non-exact, indirect determination of aluminium content, influenced by the yield of recovery, or in other words, by the quality of the recycling process. Namely,

the recovery of aluminium from the same pressed skulls performed in different recycling plants will result in different metal yields and hence different valuations of pressed skulls. Therefore, it would be very convenient and useful to have a method of fast and cost-effective non-destructive measurement of the free aluminium content in pressed skulls, influenced only by the actual pressed skulls composition. Beside simplicity and low-cost, as mentioned before, the fundamental advantage of such a measurement would be determination of the aluminium content in pressed skulls independently of the method of pressed skulls recycling. Such a methodology which could be routinely applied for measuring aluminium content in pressed skulls immediately after pressing, would also be very useful for the overall aluminium mass balance analysis in the plant and, in addition, for continuous monitoring of the quality of the dross skimming and pressing operation.



Fig. 2. An industrial device for pressing skimmed aluminium dross consisting of (1) the pressing head and (2) the pressing mould.

A functional relationship between aluminium dross bulk density and aluminium content was first reported by Manfredy et al [3] in 1997. In 2001 Norsk Hydro engineers applied for a patent for analysing aluminium in dross based on measurement of dross density [4, 5]. The theoretical background of non-destructive prediction of free aluminium content in pressed skulls, based on pressed skull density measurement, and the promising results of some experimental measurements were reported in 2002 [6]. The method was based on a linear relationship between the bulk density of pressed skulls, ρ , and metal content, w_{Al} . The linear relationship was predicted theoretically and also confirmed experimentally [6].

In the model developed at that time the amount of closed porosity in pressed skull was assumed to be negligible. However, a more detailed investigation of the microstructure of pressed skulls revealed the presence of a significant fraction of closed porosity. Hence, the purpose of this work was to improve the existing model of pressed skulls by including the volume fraction of closed pores which should be considered during aluminium content measurement. According to the new model developed, it was evident that the practical measurement of aluminium content in different pressed skulls would be possible only under well defined and considered circumstances. The most important practical consequence of the new model was classification of pressed skulls based on aluminium alloy composition and processing parameters, as well as on the closed porosity of the non-metallic phase. Finally, for industrial application of the aluminium content measuring methodology it was also necessary to predict the relative error in determining the aluminium content as the sum of relative errors in measuring the model's main variables (density of pressed skulls and non-metallic phase).

Experimental

The microstructural features (particularly the amount and morphology of open and closed pores) and chemical composition of pressed skull samples was analyzed by optical and scaning electron microscope (SEM) equipped with energy dispersive spectroscope (EDS).

Results

The macro- and microstructure of pressed skulls

Regarding macrostructure, pressed skulls consist of an **aluminium alloy shell** and a **non-metallic core**, Figs. 3-5. The aluminium alloy shell is formed during pressing of aluminium dross and squeezing out of molten aluminium alloy, appearing as a more or less continuous metallic skin covering the concave surface of the skull. During pressing of dross, this surface is in contact with the pressing mould. In contrast, the convex surface of the skull, which during pressing is in contact with the pressing head, is not covered by a metallic skin but consists of pressed non-metallic phase infiltrated with aluminium alloy.



Fig. 3: The pressed skull obtained by the pressing operation.



Fig.4. A storage area with a single class of pressed skulls. The pressed skulls inside the same class have the same alloy composition, the same density of non-metallic phase and the same volume fraction of closed porosity.

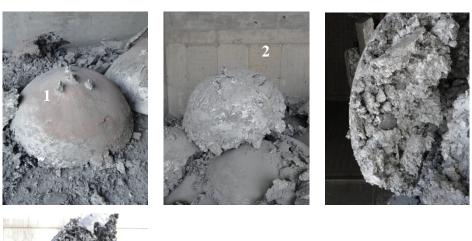




Fig. 5. The typical, non-uniform structure of pressed skulls. Pressed skulls are usually covered with an almost continuous aluminium alloy layer (1), the so-called aluminium alloy skin. Inside the pressed skull is the dominant non-metallic phase (2), heterogeneously impregnated with various amounts of discontinuously distributed aluminium alloy.

From the morphological and compositional differences in the concave and the convex surfaces of the pressed skulls it is evident that pressing of aluminium dross and squeezing out of liquid aluminium alloy introduces a strong compositional gradient inside the pressed skulls. Evidently, the concentration of aluminium alloy is the highest on the concave surface and the non-metallic phase on the convex surface of the pressed skull, as illustrated in Fig. 6.

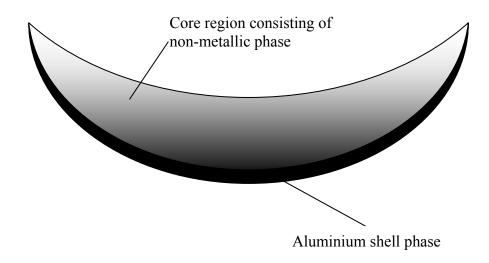


Fig. 6. Schematic representation of the macrostructure of a pressed skull

Characteristic microstructures of different regions of pressed skulls are presented in Figs. 7-10.

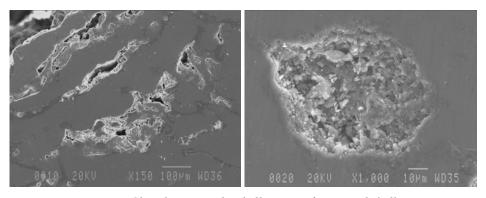


Fig. 7. Closed pores in the shell region of a pressed skull

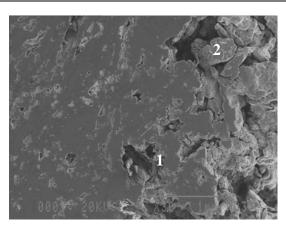


Fig. 8. Closed (1) and open (2) porosity in the shell region of a pressed skull

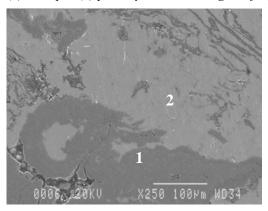


Fig. 9. SEM micrograph of the non-metallic region (1) infiltrated with alloy phase (2). Although some closed pores are present in the non-metallic phase, most of the closed porosity is located inside the aluminium alloy phase and at the interface region.

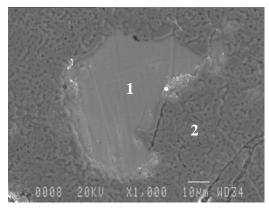


Fig. 10. The isolated aluminium region (1) surrounded by non-metallic phase (2) consisting of Al_2O_3 , MgO and spinel.

The main phases representing the major constituents of pressed skulls are the aluminium alloy phase, the non-metallic phase, and open and closed porosity.

Aluminium alloy and non-metallic phases appear in both continuous (as matrix) and discontinuous (as particulate) forms. In the shell region, an aluminium alloy continuous phase acts as the matrix, while in the core region the matrix consists of non-metallic phase. The open and closed pores are distributed in both the aluminium alloy and non-metallic phase. A detailed investigation of the distribution of open and closed pores (Figs. 7-10) in samples taken from different regions of the pressed skulls revealed that closed porosity was mostly concentrated in the aluminium phase and at the interface between the aluminium alloy and non-metallic phase. The volume fraction of closed pores varied between 5 to 40 vol. %, and was proportional to the aluminium content in the pressed skulls. In contrast, most of the open porosity was found in the non-metallic phase, in the core region of the pressed skulls.

Model of pressed skulls

As we have seen, pressed skulls consist of three main phases: free aluminium, non-metallic phase and closed porosity. The open pores, also present in pressed skulls, are beyond the scope of the present model because they do not influence the density of pressed skulls measured by the pyknometer.

Denoting the volume of closed pores in the aluminium phase as $V_{p,\text{Al}}$ and the volume of closed porosity in the non-metallic phase as $V_{p,\text{nm}}$, the volume of pressed skulls can be expressed as the following sum:

$$V = V_{Al} + V_{nm} + V_{p,Al} + V_{p,nm}$$
 (1)

The mass of a pressed skull is determined by Equation 2:

$$m = m_{Al} + m_{nm} \tag{2}$$

Hence, the density of a pressed skull is now formulated as:

$$\rho = m/V = (m_{Al} + m_{nm})/(V_{Al} + V_{nm} + V_{p,Al} + V_{p,nm}) = \rho_{Al}w_{Al} + \rho_{nm}w_{nm}$$
(3)

Considering that:

$$W_{Al} + W_{nm} + W_{p,Al} + W_{p,nm} = 1 (4)$$

$$W_{AI} + W_{nm} + p_{AI} + p_{nm} = 1 (4')$$

$$W_{AI} + W_{nm} + p = 1$$
 (4")

where $w_{p,Al} = p_{Al}$ and $w_{p,nm} = p_{nm}$ are the corresponding volume fractions of closed porosity in aluminium and the non-metallic phase, mathematically defined as:

$$W_{p,Al} = p_{Al} = V_{p,Al}/V_{Al}$$
(5)

$$W_{p,nm} = p_{nm} = V_{p,nm}/V_{nm}$$
 (6)

and

$$p = p_{Al} + p_{nm} \tag{7}$$

then, finally, expressing w_{nm} from Equation 4":

$$\mathbf{w}_{\text{nm}} = 1 - \mathbf{w}_{\text{Al}} + \mathbf{p} \tag{8}$$

and from Equation 3, one obtains:

$$\rho = \rho_{Al} w_{Al} + \rho_{nm} (1 - w_{Al} - p) \tag{9}$$

and the volume fraction of aluminium:

$$\mathbf{w}_{Al} = [(\rho_{nm}(1 - \mathbf{p}) - \rho)]/(\rho_{nm} - \rho_{Al}) \tag{10}$$

as the function of the density of the pressed skull, ρ , the theoretical density of the aluminium phase, ρ_{Al} , the theoretical density of the non-metallic phase, ρ_{nm} , and the volume fraction of closed pores in the pressed skull, p.

Finally, by assuming that the closed porosity, p, in pressed skulls is inversely proportional to the aluminium content and expressing that by a decreasing linear function of the aluminium content, w_{Al} :

$$p = p_0 (1 - w_{Al})$$
 (11)

where p_0 is the closed porosity of pressed skulls with $w_{Al} = 0$ (i.e. the closed porosity of aluminium-free non-metallic phase). By combining Eq. (11) and Eq. (10), the following relation between the aluminium content, w_{Al} , and the pressed skulls density, ρ , can be derived:

$$\mathbf{w}_{Al} = [\rho - \rho_{nm} (1 - \mathbf{p}_0)] / [\rho_{Al} - \rho_{nm} (1 - \mathbf{p}_0)]$$
(12)

Finally, by rearranging Equation 12, the volume fraction of aluminium in pressed skulls can be expressed as a linear function of the pressed skulls density:

$$\mathbf{w}_{\mathsf{A}\mathsf{I}} = \mathsf{k}\rho + \mathsf{n} \tag{13}$$

where parameters k and n are defined as follows:

$$k = 1/[\rho_{Al} - \rho_{nm}(1 - p_0)] \tag{14}$$

$$n = -\rho_{nm}(1 - p_0)/[\rho_{Al} - \rho_{nm}(1 - p_0)]$$
(15)

Equations 14 and 15 clearly demonstrate that parameters k and n are dependent on the aluminium alloy density, as well as the non-metallic phase density and closed porosity. Moreover, from Equations 13-15 is evident that a practical evaluation of the aluminium content in pressed skulls based on its density is possible only with a knowledge (calculated or experimentally determined) of the constants k and n. Based on Equations (14) and (15), this would be the case as long as the density and closed porosity of the non-metallic phase as well as the density of the aluminium alloy remain constant. If these conditions are not fulfilled, evaluation of the aluminium content in pressed skulls based on density is not possible. For example, by using Equations 12 and 13 it is easily to recognize that if k and n are not constant (i.e. the aluminium alloy density, the density of the non-metallic phase and the volume fraction of closed pores are not the same), the functional relationship between aluminium content and pressed skull density becomes ambiguous. In other words, different densities of pressed skulls might correspond to the same aluminium content or vice versa, making evaluation of free metal impossible.

The relative error of prediction of aluminium content in pressed skulls

To determine the aluminium content in pressed skull within the desired relative error, $RE(w_{Al})$, the relative error in measuring the density of pressed skulls, $RE(\rho)$, the density of the non-metallic phase, $RE(\rho_{nm})$, and the closed porosity of the non-metallic phase, $RE(\rho_0)$, should be lower than their critical values.

The relative errors of particular variables present in Equation 12 are defined as:

$$RE(w_{Al}) = dw_{Al}/w_{Al} \tag{16}$$

$$RE(\rho) = d\rho/\rho \tag{17}$$

$$RE(\rho_{nm}) = d\rho_{nm}/\rho_{nm}$$
 (18)

$$RE(p_0) = dp_0/p_0 \tag{19}$$

From the derivation of Equation 12 and by appropriate mathematical transformations, the following final expression can be derived:

$$RE(w_{A1}) = [1/[\rho - \rho_{nm}(1 - p_0)]]RE(\rho) + [\rho_{nm}(\rho - \rho_{A1})/[(\rho_{A1} - \rho_{nm}(1 - p_0))(\rho - \rho_{nm}(1 - p_0))]$$

$$[p_0)$$
] RE $[p_0]$ + $[(1-p_0)(\rho - \rho_{Al})/[(\rho_{Al} - \rho_{nm}(1-p_0))(\rho - \rho_{nm}(1-p_0))]$ RE $[\rho_{nm}]$ = K_1 RE $[\rho]$ +

$$K_2 RE(p_0) + K_3 RE(\rho_{nm})$$
(20)

where:

$$\mathbf{K}_1 = \partial \mathbf{w}_{\text{Al}}/\partial \rho = 1/[\rho - \rho_{\text{nm}}(1 - \mathbf{p}_0)] \tag{21}$$

$$K_2 = \partial w_{Al}/\partial p_0 = \rho_{nm}(\rho - \rho_{Al})/[(\rho_{Al} - \rho_{nm}(1 - p_0))(\rho - \rho_{nm}(1 - p_0))]$$
(22)

$$K_3 = \partial w_{Al}/\partial \rho_{nm} = (1 - p_0)(\rho - \rho_{Al})/\left[(\rho_{Al} - \rho_{nm}(1 - p_0))(\rho - \rho_{nm}(1 - p_0)) \right]$$
(23)

Equation 20 enables prediction of the relative error of determination of aluminium content in pressed skulls based on the known relative errors in determining

the density of pressed skulls, the density of the non-metallic phase and the closed porosity in the non-metallic phase of pressed skulls.

Let us consider, for example, that the density of pressed skulls is experimentally measurable within a relative error of 0.1%, and the density as well as the volume fraction of closed porosity in the non-metallic phase within a relative error of 1%. In addition, it can also be assumed that the closed porosity in aluminium-free ($w_{Al}=0$) non-metallic phase is 20 vol. %. For that particular case, the calculated relative error in determining the aluminium content in pressed skulls with different aluminium content (from 10 to 90 vol. %) is presented in Table 1. The calculation is made under the assumption that the closed porosity (p_0) in the aluminium-free non-metallic phase ($w_{Al}=0$) is 20%.

As evident, the relative error in determining the aluminium content in pressed skulls with an aluminium content below 30 vol. % ($w_{Al} < 0.3$) is unacceptably high. However, the relative error in determining the aluminium content in pressed skulls with an aluminium content higher than 30 vol. % ($w_{Al} > 0.3$) is fully acceptable for industrial applications. Moreover, considering that the density of pressed skulls can be experimentally determined with an approx. one order of magnitude lower relative error than the density and the closed porosity of the non-metallic phase, the coefficient K_2 Re(ρ_{nm}) involving the relative error in measuring the density of the non-metallic phase is the dominant contribution to the relative error in determining the aluminium content, contributing more than 75%.

Table 1 The calculated relative error in determining the aluminium content, w_{Ab} in pressed skulls as a function of pressed skull density, density of the non-metallic phase and their corresponding relative errors.

W_{Al}	ρ	$ ho_{ m nm}$	P_0	K_1	K_2	K_3	dw_{Al}/w_{Al} , %
							$d\rho/\rho = 0.001$
							$d\rho_{nm}/\rho_{nm}=0.01$
							dp/p = 0.01
0.1	3.48	4.46	0.2	11.4	45.6	8.2	54.9
0.2	3.39	4.46	0.2	5.6	19.9	3.6	24.0
0.3	3.31	4.46	0.2	3.9	12.1	2.2	14.7
0.4	3.22	4.46	0.2	2.9	7.7	1.4	9.4
0.5	3.13	4.46	0.2	2.3	5.0	0.9	6.1
0.6	3.05	4.46	0.2	1.9	3.5	0.6	4.3
0.7	2.96	4.46	0.2	1.6	2.2	0.4	2.8
0.8	2.87	4.46	0.2	1.4	1.3	0.2	1.6
0.9	2.79	4.46	0.2	1.3	0.6	0.1	0.8

Application of the model in industrial practice

Determination of parameters k and n

Calculation of parameters k and n from experimentally determined data

Theoretically, the parameters k and n appearing in Equation (13) can be calculated by using Equations (14) and (15), based on the experimentally determined density of the aluminium alloy (ρ_{Al}), the density of the non-metallic phase (ρ_{nm}) and the volume fraction of closed porosity (ρ_0) in the non-metallic phase.

Measurement of the pressed skulls density

Measurement of the pressed skulls density is performed by a specially designed industrial pyknometer with a pressure vessel large enough to enable measurement of the entire pressed skull volume without sub-sampling.

The principle of the pressed skulls volume measurement is based on the ideal gas equation:

$$pV_{v} = nRT \tag{24}$$

applied first to the empty pressure vessel and then to the pressure vessel containing the pressed skull.

In Equation (24) p, V_v and T represent the pressure, volume and number of moles of gas introduced into the pressure chamber, while R is the universal gas constant.

At constant temperature and the same number of moles of gas introduced into the vessel in both cases, the following relation can be written:

$$P_1 V_v = p_2 (V_v - V) \tag{25}$$

in which p_1 is the pressure of the gas measured in the empty vessel, p_2 is the pressure of gas in the vessel containing pressed skulls and V is the volume of pressed skulls consisting of the volume of aluminium, non-metallic phase and closed porosity.

Finally, by solving equation 25 the volume of pressed skulls can be expressed as a function of routinely measurable variables:

$$V = V_{\nu} \left(\Delta p / p_2 \right) \tag{26}$$

where $\Delta p = p_2 - p_1$.

Regarding the accuracy of the measurement, it is important to note that the method described is absolutely capable of providing determination of the density of pressed skulls within a relative error of $\pm 0.05\%$, operating with a relative error of mass measurement of $\pm 0.02\%$ (± 50 g) and a relative error of measurement of the volume of pressed skulls of $\pm 0.03\%$ (± 25 cm³).

Measuring the density of the non-metallic phase

In contrast to daily industrial determination of the density of pressed skulls, the density of the non-metallic phase is strictly a laboratory analysis, performed occasionally, mainly for the proper classification of different types of pressed skulls into separate classes, as discussed earlier. The density of the non-metallic phase (pure non-metallic phase without aluminium), as presented in the particular class of pressed skulls, is measured with a prescribed number (12-25) and mass (1 kg) of representative subsamples, using a laboratory pyknometer. It is important to note that the number and size of sub-samples are usually selected to represent about 10% of the total amount of non-metallic phase in the pressed skulls.

Before density measurement, the 1 kg sub-samples were milled in a planetary mill in order to obtain a uniform composition, without aluminium, which is separated from the non-metallic fraction by sieving, and to eliminate possible closed porosity inside particle aggregates and clusters. The absence of aluminium in the sieved powder is additionally confirmed by wet chemical analysis of randomly selected sub-samples (dissolution of any present traces of aluminium in HCl and analysing the solution obtained by ICP or other alternative methods).

A small portion (5-10 g) of the milled and well-homogenized aluminium-free powder, usually having an average particle size below 10 μ m, is then applied for final density measurement of the non-metallic phase. The accuracy of an individual density measurement is about $\pm 0.01\%$, and the relative error of predicting the density of the non-metallic phase on the volume level of pressed skulls, taking into consideration measurement of the density of all sub-samples, is about $\pm 0.25\%$, which is far below the required $\pm 0.5\%$ (see Table 1).

The relative error of an individual density measurement was calculated using the following formula:

$$d\rho/\rho = (dm/m) - (dV/V^2) \tag{27}$$

Here, m represents the mass of pressed skulls and V its volume measured by the piknometer.

Measuring the closed porosity in the non-metallic phase

In principle, the closed porosity of the non-metallic phase can be determined by measuring the density of representative samples of the as-received non-metallic phase $(\rho_{nm,p})$ and the density of representative samples of the milled non-metallic phase $(\rho_{nm,0})$, which is free of closed porosity:

$$P_0 = I - (\rho_{nm,p}/\rho_{nm,0}) \tag{28}$$

The relative error of the closed porosity measurement is defined by the following relation:

$$dp0/p0 = -d \rho nm, 0/(\rho nm, p - \rho nm, 0) + [(\rho nm, 0/\rho nm, p)/(\rho nm, p - \rho nm, 0)]d \rho nm, p (29)$$

Taking into consideration the usual values of the non-metallic phase density $(\rho_{nm,p}=3.00g/cm^3,~\rho_{nm,0}=3.75~g/cm^3)$ and closed porosity $(p_0=0.2)$, as well as the

absolute error in determining the density (d $\rho_{nm,0} = d \rho_{nm,p} = 2.5 \times 10^{-3}$), one can calculate that the relative error in determining the closed porosity of *individual* non-metallic phase samples does not exceed 1% ($\pm 0.5\%$).

However, practical measurement of the closed porosity in the non-metallic phase of pressed skulls within the required accuracy of $\pm 0.5\%$ is very demanding and cannot be performed on an industrial scale. The problem is not in the measurement technique but in providing a sufficient amount of aluminium-free species of the non-metallic phase. In order to reach $\pm 0.5\%$ accuracy of the closed porosity measurement, at least 10% (10-15 kg) of the mass of non-metallic phase present in pressed skulls should be representatively sampled and analysed. In practice, larger samples of the non-metallic phase are always contaminated with aluminium. In this respect, for collecting aluminium-free samples of the non-metallic phase, the mass of individual samples should be kept small enough (usually 20-50 g). Such a sampling procedure however creates a very large number of samples to be analysed, which is unacceptable for an industrial approach.

Determination of parameters k and n from the graph w_{Al} - ρ

As explained earlier, the parameters k and n of the linear function $w_{Al} = k\rho + n$ cannot be calculated with sufficient accuracy using the experimentally determined values of ρ , ρ_{nm} and p_0 . Hence, the practical way of determining parameters k and n is from the experimentally plotted graph – by measuring the density of pressed skulls (ρ) (as described in Section 4.1.1.1) and the corresponding volume percentage of recovered aluminium (w_{Al}).

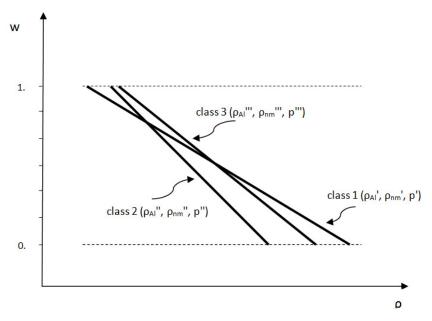


Fig. 11. Set of linear graphs corresponding to different classes of pressed skul

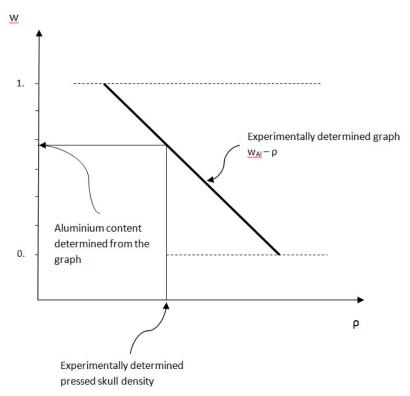


Fig. 12. Method of determining the aluminium content in pressed skulls based on the measured density of skulls and the graph $w_{Al} - \rho$, plotted according to the experimentally collected data on pressed skulls density and aluminium recovery by melting.

Conclusion

OM and SEM/EDS investigation of the microstructure of both shell and core regions of pressed skulls showed that the prevailing amount (up to 40 vol. %) of closed porosity is in the aluminium shell region and of open porosity in the core region.

Because of that, in the new model of pressed skulls developed in this work, closed porosity was involved as one of the main components, along with aluminium and the non-metallic phases.

Based on that, the aluminium content in pressed skulls was functionally correlated with pressed skulls density and the other parameters (aluminium alloy density, non-metallic phase density and the volume fraction of closed porosity in pressed skulls). In addition, by postulating that the amount of closed porosity is inversely proportional to the aluminium content, the closed porosity of pressed skulls was expressed in the final model by the closed porosity of the non-metallic phase.

Under conditions when other parameters except the pressed skulls density remain constant, the aluminium content in pressed skulls can be expressed by the linear function $w_{Al} = k\rho + n$ in which parameters k and n are constants depending on the aluminium alloy density, the non-metallic phase density and the porosity of the non-

metallic phase. However, in general, the aluminium content in pressed skulls is a rather complex function of all the above mentioned variables, as proved by the model.

Theoretical analysis of the relative error of determination of the aluminium content in pressed skulls performed in accordance with the new model demonstrated that in skulls with an aluminium content below 40 vol. %, the relative error is unacceptably high, while in skulls with an aluminium content higher than 40 vol. % it is within the acceptable $\pm 5\%$. Note that these results were calculated for the case that the relative error of pressed skulls density does not exceed $\pm 0.05\%$ and the relative errors in the non-metallic phase density as well as in the volume fraction of closed pores are less than $\pm 0.5\%$.

References

- [1] Hong Jian-Ping, Wang Jun, Chen Hai-Yan, Sun Bao-De, Li Jia-Jing and Chen Chong, Trans. Nonferrous Met. Soc. China, 20 (2010) 2155-2161
- [2] A. M. Peel, J. Herbert, D. Roth and M. J. Collins, Materials Science Forum, Vol. 630 (2010) 53-60
- [3] O. Mafredi, W. Wuth and I. Bohlinger, JOM, 47 (1997) 11, 48-51
- [4] US Patent Application 20110197670, 2009
- [5] WIPO Patent Application WO/2001/020300
- [6] V. Kevorkijan, JOM, 54 (2002) 2, 34-36