The seventh LBMA Assaying and Refining Conference

A report after one year of experimentation and real production

presented by Philippe Cettou
ALS in conferences & seminars

For the last two years, ALS process has been presented in many conferences & seminars, for example:

• ALS @ LBMA in 2015 (London)
• ALS @ IPMI, in 2015 (San Antonio) and 2016 (Phoenix)
• ALS @ Jewellery Technology Forum in 2016 (Vicenza)

A very informative article was also published in the Alchemist:

Acidless Separation: New technology for refining gold and silver,
Giovanni Faoro, CEO IKOI Srl, Alchemist, Issue 79, Oct 19, 2015
Let me first remind you what is ALS!

- ALS is a pre-refining process removing silver from Au/Ag/Cu alloys using vacuum distillation technology.

- It is used to reduce silver content on Au/Ag/Cu alloys, in order to make the resulting gold based alloy suitable for treatment in the subsequent final refining step (like Wohlwill gold electrolysis).

- The other usual elements that can be found in PM feedstock to be treated will either be distilled or not, depending on their vapour pressure characteristics: ALS will remove zinc, lead and other high volatile components.
ALS – How does it work?
**Furnace description**

- **Inductor**: From 50 to 150 kW power
- **Vacuum chamber with water cooled double wall**
- **Melting zone**: Up to 1450°C Continuous recording of temperature
- **Crucible**: From 30 kg to 100 kg alloy capacity
- **Weight measurement system**: Continuous recording of crucible weight during the process
- **Condenser for Ag and other light elements**
The ACIDLESS SEPARATION® plant comes from the collaboration of two companies:

EZ OCM-Engineering LLC  
Refining, processing, research & development and production of industrial items made of gold, silver and PMGs

IKOI Srl  
Machines, plants and technologies for melting, heating, chemical and physical treatments of precious metals
The vapour pressure of metallic elements

The process uses the properties of each element showing a specific vapour pressure at a specific temperature.

The literature provides a table giving vapor pressure data as a function of temperature, following the simplified Clausius-Clapeyron relation:

\[ \ln p = A - B/T \]

For a convenient performance, the conditions chosen for ALS are:

- a pressure around $10^{-2} - 10^{-3}$ mbar
- and a temperature from 1300°C up to 1450°C.
Selectivity of the separation

Separation coefficient $K$

$$K = \frac{P_1(T)}{P_2(T)} \frac{x_1(T)}{1-x_1(T)}$$

But note that when the molar fraction of the most volatile element tends to zero, then the $K$ value strongly decreases.

As a consequence, the selectivity decreases when the molar fraction of a particular element becomes low.

Empirical rule to evaluate the selectivity in the separation process:

$$K > 100$$

As an example for:

Au 70% Ag 30% at $T=1'350°C$

$$K \approx 150$$
Ideal rate of evaporation as a function of temperature is given by Langmuir’s equation:

\[
\dot{m} = \frac{p_v}{\sqrt{2\pi MRT}}
\]

- \( M \) = molar mass of the element
- \( R \) = ideal gas constant
- \( T \) = absolute temperature
- \( P_v \) = vapor pressure of the element

Example:

\[ T = 1'350 \, ^\circ\text{C} \]
\[ \text{Crucible diameter} = 20 \, \text{cm} \]

\[ \dot{m}_{\text{Ag}} = 315 \, \text{g/min} \]
\[ \dot{m}_{\text{Au}} = 1.6 \, \text{g/min} \]
\[ \dot{m}_{\text{Cu}} = 10 \, \text{g/min} \]
Process simulation

Variation of elements mass vs time

$m/m_0$

Evaporation step 1
Volatile impurities

Evaporation step 2
Silver

Time [min]

Au
Ag
Zn
Se
Pb
Cu
Example #1: Binary alloy Au/Ag

Input: Binary alloy Au/Ag
- Mass: 16'749 g
- Composition:

<table>
<thead>
<tr>
<th>%Au</th>
<th>%Ag</th>
</tr>
</thead>
<tbody>
<tr>
<td>49.2</td>
<td>50.8</td>
</tr>
</tbody>
</table>

ALS
Tev=1'450°C
tev=70 min

Prerefinned billet
- Mass: 8'465 g
- Composition:

<table>
<thead>
<tr>
<th>%Au</th>
<th>%Ag</th>
</tr>
</thead>
<tbody>
<tr>
<td>97.2</td>
<td>2.8</td>
</tr>
</tbody>
</table>

Condensate
- Mass: 8'274 g
- Average vaporation rate = 120 g/min
- Composition:

<table>
<thead>
<tr>
<th>%Au</th>
<th>%Ag</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.7</td>
<td>97.3</td>
</tr>
</tbody>
</table>
Process steps for example #1 - Binary alloy Au/Ag

Temperature & Mass vs Time

Temperature [°C] vs Mass [g]

HEATING & MELTING
HEATING
EVAPORATION
COOLING

0:00 0:20 0:40 1:00 1:20 1:40 2:00

0 200 400 600 800 1000 1200 1400 1600 1800 2000

0 2000 4000 6000 8000 10000 12000 14000 16000 18000 20000
Example #2: Doré with base metals

Input: Doré with base metals
- Mass: 7'800 g
- Composition:

<table>
<thead>
<tr>
<th>%Au</th>
<th>%Ag</th>
<th>%Zn</th>
<th>%Se</th>
<th>%Cu</th>
<th>%Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>49</td>
<td>35</td>
<td>6</td>
<td>4</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

Prerefined billet
- Mass: 4'230 g
- Composition:

<table>
<thead>
<tr>
<th>%Au</th>
<th>%Ag</th>
<th>%Zn</th>
<th>%Se</th>
<th>%Cu</th>
<th>%Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>89,8</td>
<td>5,3</td>
<td>-</td>
<td>-</td>
<td>4,9</td>
<td>-</td>
</tr>
</tbody>
</table>

ALS STEP 1
Tev=1'200°C
tev=20 min

Condensate from STEP 1
- Mass: 1'150 g
- Average evaporation rate = 57.5 g/min
- Composition:

<table>
<thead>
<tr>
<th>%Au</th>
<th>%Ag</th>
<th>%Zn</th>
<th>%Se</th>
<th>%Cu</th>
<th>%Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>0,1</td>
<td>10,5</td>
<td>39,3</td>
<td>23,9</td>
<td>0,3</td>
<td>25,9</td>
</tr>
</tbody>
</table>

ALS STEP 2
Tev=1'480°C
tev=40 min

Condensate from STEP 2
- Mass: 2'420 g
- Average evaporation rate = 60,5 g/min
- Composition:

<table>
<thead>
<tr>
<th>%Au</th>
<th>%Ag</th>
<th>%Zn</th>
<th>%Se</th>
<th>%Cu</th>
<th>%Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>0,8</td>
<td>96,4</td>
<td>-</td>
<td>-</td>
<td>2,8</td>
<td>-</td>
</tr>
</tbody>
</table>
Process steps for example #2: Doré with base metals

Temperature and mass vs time

Mass [g]

Temperature [°C]

0:00 0:15 0:30 0:43 0:58 1:13 1:28

HEATING & MELTING  EVAPORATION 1  EVAPORATION 2
ALS Scale - up
ALS in the world of PM refiners:

- ALS30 in production since March 2015 in Russia
- ALS30 in production since May 2016, in Kazakhstan
- ALS60 to be delivered to PAMP SA/Switzerland early 2017
- ALS60 to be delivered to ARGOR-HERAEUS SA/Switzerland March 2017
- ALS100 ordered by ABC REFINERY/Australia, to be delivered in a few weeks
What has been done so far with ALS?
(upto 31.12.2016)

- **ALS 30**
  - Russia
  - In production since March 2015
  - 4'800 kg

- **ALS 30**
  - Kazakhstan
  - In production since May 2016
  - 3'000 kg

- **ALS 60**
  - Italy
  - For demonstration and testing different customer alloys
  - 700 kg

ALS: a report after one year of experimentation and real production
How much silver has been evaporated with ALS up to now? (until 31.12.2016)

Input: Doré
Mass: 8'556 kg
Silver: 1'616 kg
%Ag: 19%

Output: Condensate
Mass: 1'515 kg
Silver: 1'337 kg
%Ag: 86%

Output: Billet
Mass: 7'002 kg
Silver: 281 kg
%Ag: 4%
How selective is ALS?

In absolute values

In relative values

- In red: what is left in the billet
- In green: what is found in condensate
Main advantages of ALS technology

- **GREEN TECHNOLOGY**: No use of chemicals
- **FAST**: Compared to other pre-refining steps, the average residence time is low
- **LOW OPERATING COST**: Very limited manual operation
- **UNIVERSAL**: ALS process can be used on a large panel of PM based alloys
- **SAFE & USERFRIENDLY**: This batch process takes place in an enclosed volume and is fully automatized. No PM’s losses
ACIDLESS SEPARATION® PROCESSING

This is an ALS® plant (ALS30)
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Open the chamber
ACIDLESS SEPARATION® PROCESSING

Insert the feedstock into the crucible: doré bar or ...
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...pre-homogenized billet

ALS: a report after one year of experimentation and real production
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Close the chamber
Recall a recipe, or setting up a new one, and start the process.
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Vacuum creation → heating → melting → evaporation → cooling down
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The process is finished: cut vacuum and open the chamber
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This is the billet in the crucible, rich in gold
ACIDLESS SEPARATION® PROCESSING

Taking out the billet
ACIDLESS SEPARATION® PROCESSING

The billet, rich in gold
ACIDLESS SEPARATION® PROCESSING

Taking out the condenser
ACIDLESS SEPARATION® PROCESSING

This is the inside of the condenser, showing the silver rich condensate.
ACIDLESS SEPARATION® PROCESSING

The material, which has the consistency of a dry mud, can be easily removed
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This is the removed condensate, rich in silver
ACIDLESS SEPARATION® PROCESSING

The result of the ALS separation process:
the billet rich in gold, and the condensate rich in silver
a very promised process for PM’s refiners!

Do you want to be at the forefront of technology?
Are you aiming to be seen as having a real concern for green technology?
Do you want to offer clean and safe working conditions to your employees?
Are you interested in saving costs on pre-refining?

If YES, it’s time for ALS!
Interested?

GTF GREEN TECHNOLOGY & FINANCE,
worldwide distributor for the ALS® technology,
offers you formulas as innovative and disruptive as ALS.
Thank you for your attention!

*All mentioned data and pictures in this presentation are not binding and are only used, as example, for commercial purpose

www.ikoi.it  www.gtf-uk.com
ALS 60

ALS: a report after one year of experimentation and real production
ALS 60