CHALLENGES IN THE MANUFACTURING OF SILVER REFERENCE MATERIALS

I.B. Khobyakova, P.A. Khorikov, D.R. Shulgin
K.A. Shatnykh, V.N. Efimov, S.N. Mamonov
Krastsvetmet JSC (Russia)
THE OUTLINE OF THE PRESENTATION

- Manufacturing of the material for reference materials production
- Assaying
- Conclusions

The Reference Material Composition
Ag RM 1, Ag RM 2

<table>
<thead>
<tr>
<th>Element</th>
<th>Ag RM 1</th>
<th>Ag RM 2</th>
</tr>
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<tbody>
<tr>
<td>Al</td>
<td>5-10</td>
<td>20-40</td>
</tr>
<tr>
<td>As</td>
<td>20-40</td>
<td>5-10</td>
</tr>
<tr>
<td>Au</td>
<td>5-10</td>
<td>20-40</td>
</tr>
<tr>
<td>Bi</td>
<td>10</td>
<td>50</td>
</tr>
<tr>
<td>Cd</td>
<td>5-10</td>
<td>20-40</td>
</tr>
<tr>
<td>Cr</td>
<td>5-10</td>
<td>20-40</td>
</tr>
<tr>
<td>Cu</td>
<td>20</td>
<td>100</td>
</tr>
<tr>
<td>Fe</td>
<td>5-10</td>
<td>20-40</td>
</tr>
<tr>
<td>Mg</td>
<td>20-40</td>
<td>5-10</td>
</tr>
<tr>
<td>Mn</td>
<td>5-10</td>
<td>20-40</td>
</tr>
<tr>
<td>Ni</td>
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<tr>
<td>P</td>
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<td>20-40</td>
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<tr>
<td>Pd</td>
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<td>20-40</td>
</tr>
<tr>
<td>Pt</td>
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<tr>
<td>Rh</td>
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<tr>
<td>Sb</td>
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<td>Se</td>
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<tr>
<td>Si</td>
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<td>Te</td>
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<td>20-40</td>
</tr>
<tr>
<td>Zn</td>
<td>20-40</td>
<td>5-10</td>
</tr>
</tbody>
</table>

Fig. 1. Slight chamfering due to guillotining

Fig. 2. Other edge after guillotining
The PRINCIPAL FLOW CHART
Ag RMs manufacturing

Stage 1
Selecting of the initial basic material
Cycle Start

Stage 2
Master alloys melting
Composition analysis

Stage 3
Pre-melting of RM's metal
Composition analysis

Stage 4
Combined melting of the RM's metal
Composition and homogeneity analysis

Cycle Start

1. Selecting of the initial basic material
2. Master alloys melting
3. Pre-melting of RM's metal
4. Combined melting of the RM's metal

The starting metals to produce the Ag RMs

Refined Silver granules

- Merck and Alfa additive metals
MASTER ALLOYS MANUFACTURE

- As a result of melting 20 master alloys of cylindrical form were produced:
  Ag-Al, Ag-As, Ag-Au, Ag-Bi, Ag-Cd, Ag-Cr, Ag-Cu, Ag-Mg, Ag-Mn, Ag-Ni, Ag-Pb, Ag-Pt, Ag-Rh, Ag-Sb, Ag-Se, Ag-Si, Ag-Sn, Ag-Te, Ag-Zn, Ag-Pd-Fe

PREMELTING

- The inside of Supercast 5000 VAC Induction Furnace with the casting in the mould.

- The appearance of the casting:
  - as-cast;
  - After surface milling
Combined Casting into a Horizontal Iron-cast Mould

Results of homogeneity testing of the metal cast into a horizontal iron-cast mould
Combined melting and casting into a book-type (vertical) iron-cast mould

- Vertical iron-cast mould
- The inside was sprayed coated with magnesium suspension
- The appearance of the vertical ingot

Annealing and milling of the casting

- The furnace used for annealing before rolling the bars
- Milling the annealed bars before rolling
The rolling mill

Rolled material marking - preparation for homogeneity testing

- Marking of the rolled material;
- The square pieces resulted from the rolled material cutting (26x26x7 mm)
### Homogeneity Testing

<table>
<thead>
<tr>
<th>Ag RM1</th>
<th>Homogeneity Test (in-plant)</th>
<th>Homogeneity Test (in-plant)</th>
</tr>
</thead>
<tbody>
<tr>
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</table>

### Preparation of samples for homogeneity testing

![Image of sample preparation process]

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The LBMA Assaying & Refining Seminar
7-8 March 2011
Testing Scheme

- Top side of the piece
- Middle (cut) side
- Bottom side

Results of Homogeneity Testing
Additional Testing for Homogeneity

Analytical support

- Testing procedures for Ag RMs Production Project by Krastsvetmet accredited laboratory equipped with both the methods and apparatus appropriate for meeting challenges with high degree of validity
- Testing of bi-metal master alloys was carried out by X-ray-spectral method using Philips PW 2404 X-Ray Fluorescent Spectrometer
Analytical support

- Chemical composition and homogeneity testing was done by Spark Atomic Emission method using ARL 4460 Optical Emission Spectrometer.
- State Reference Materials for silver were used for checking of the spectrometer for shifting at the start of measurement and then every 30 results.

Analytical support

- Analysis of Silver Shavings to certify the composition of the Ag Reference Material within inter-lab checking. The analysis was carried out by ICP-AES.

Preparation of the shavings for analysis:
- washing in hot 50% HCl;
- washing in distilled deionized water;
- drying in a vented clean hood.
Analytical support

The silver shavings were analyzed by ICP-AES.

Thermo Electron IRIS Intrepid II DUO Spectrometer was used.

Analysis Certificate

Laser etching was used for labelling Ag RM 1, Ag RM 2.
Conclusions

• As a result of the efforts a set of Silver Reference Materials has been produced: Ag RM1 and AgRM2
• Homogeneity testing was carried out by JSC Krastsvetmet and cross-checked by Rand Refinery by Spark atomic-emission optical spectrometry.
• 12 laboratories were involved in the inter-laboratory checks for certification.

Conclusion

• Perth Mint, Australia;
• Aurubis, Germany;
• Cendres+Metaux, Switzerland;
• Krastsvetmet JSC, Russia;
• Matusudo Sangyo Co Ltd, Japan;
• Metalor Technologies, Switzerland;
• Johnson Matthey, Canada;
• Rand Refinery, South Africa;
• Royal Canadian Mint, Canada;
• Sumiko Techno Research Co Ltd, Japan;
• Tanaka Kikinzoku Kogyo, Japan;
• Umicore Precious Metals Refining, Belgium.
Conclusion

• The Joint Project for Au and Ag Certified Reference Materials has been a successful example of a promising and mutually beneficial collaboration of the parties concerned in meeting the challenges of industry.

THANK YOU FOR YOUR ATTENTION