Manufacturing of Gold Reference Materials

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Hitoshi Kosai, Hiroshi Sawai
Nobuyasu Ezawa, Ichimitsu Itabashi
Tanaka Kikinzoku Kogyo K.K.
### Agenda

1. **Manufacturing of Gold Reference Materials**  
   *Manufacturing process is not special*  
   *The core process is Melting & Casting*

2. **Assaying for the Gold Reference Materials**  
   *Sampling & Assay for rough verification*  
   *Homogeneity test*  
   *Assay to determine of contents*

3. **Conclusion**

### LBMA Gold Reference Materials

<table>
<thead>
<tr>
<th>Date</th>
<th>Experiment</th>
<th>Site Visit by LBMA</th>
<th>Crucible &amp; Mold</th>
<th>Master Alloy</th>
<th>Ingot Making</th>
<th>Rolling</th>
<th>Cutting</th>
<th>Grinding</th>
<th>Marking</th>
<th>Packing</th>
<th>Shipping</th>
<th>Homogeneity Test</th>
<th>TKK Pre-Test</th>
<th>Rand Refinery Assay</th>
<th>TKK Assay</th>
<th>9 Laboratories Assay</th>
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<tbody>
<tr>
<td>Mar 08</td>
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<td>Prepare</td>
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**Progress of LBMA- RM <<Au>> producing**  
- TKK work
- LBMA Project work
- Subcontractor work

**Timeline**

- Mar...
- Aug Sep
- Apr May
- Jun Jul
- Oct Nov
- Dec Jan 09
- Feb Mar
Manufacturing Flow Chart

Pure Au (6N grade)
Assayed impurities
(Total 14kg)
Melting
Induction Furnace
Carbon Crucible
Inert gas after Vacuum
Casting
Carbon Book Shelf Mold
Au RM Ingot

Sampling & Assay for rough verification
Analyzing drilled sample from Top and Bottom of RM Ingot
Surface Shaving
Shaper Machine
Rolling
Rolls are exclusively for Au manufacture
Roll RM Ingot to specified thickness 6mm
Cutting
Cut sub-sample piece 21mm × 21mm
Grinding
One edge of pieces is grinded to Marking
Homogeneity test
Sampling on RM project’s rule (15 pieces and 6 dimensions of depth)
Both Tanaka and Rand Refinery assay
Shaving
Shavings are made from these pieces for Homogeneity test
Assay to determine of contents
By 10 laboratories which are selected by the Steering committee
Assurance of Contents and Homogeneity

Melting & Casting
Various Methods
Contents Assay
Wet ICP
Master Au Alloys

Master Alloys & 5N Pure Au
Melting & Casting 1

Vacuum Furnace

Elements Charge Bucket

Carbon Crucible

Carbon Book Shelf Mold

Inside of the vacuum furnace

Melting & Casting 2

Elements Charge

Casting

Au Ingot for LBMA RM
Surface Shaving

Before Shaving

After Shaving

Shaving by Shaper Machine

Rolling

Manual Rolling at beginning of process

Automatic two-way tandem Rolling

Rolled Plate for LBMA Au RM
Cutting & Grinding 1

Guillotine Cut to Oblong Card Form

Grinding to make Flat Marking Plane

Gutting & Grinding 2

Cutting to piece without confusing of address
Marking

Marking Template

Complete LBMA Au RM pieces

Template goes down and hits a piece

Assay of contents

1. Contents Assay for Some of Master Alloys
2. Sampling & Assay for rough Verification from RM ingot
3. Shaving
4. Homogeneity test
5. Pre-operation for wet-ICP

Manufacture

Contents Assay

Pure Au

Melting & Casting

Au-RM ingot

Surface Shaving

Rolling, Cutting, Grinding

Select 15 pieces

Shaving

Shaving Sample

Assay by 10 laboratory

Pre-operation for wet-ICP

Homogeneity Test

Pieces after Shaving

Contents Assay

Master Au Alloys

Shaving Sample
2. Sampling & Assay for rough Verification from RM ingot

<table>
<thead>
<tr>
<th>Element</th>
<th>Copper</th>
<th>Tab</th>
<th>Assay</th>
<th>Shaving</th>
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<td>Ag</td>
<td>99.70%</td>
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<tr>
<td>Au</td>
<td>0.30%</td>
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<tr>
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<td>98.7%</td>
<td>98.8%</td>
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<tr>
<td>Cu</td>
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<td>0.2%</td>
<td>0.3%</td>
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<tr>
<td>Ag</td>
<td>99.80%</td>
<td>99.4%</td>
<td>99.3%</td>
<td>99.5%</td>
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<tr>
<td>Au</td>
<td>0.20%</td>
<td>0.6%</td>
<td>0.7%</td>
<td>0.8%</td>
</tr>
</tbody>
</table>

3. Shaving

Shaving Machine

Flying Cutter

Shaving of Au-RM
4: Homogeneity test

- **Shrinkage**
- **Column**
- **Row**
  - 7 Columns
  - 19 Rows
  - 15 pieces

**Assay using Spark-Ablation ICP**

- iCAP-SSEA System (ThermoFisher Scienfitec)

- 90 measurements (15 pieces * 2 faces * 3 repeats)
- Randomize Assay (Sequence of indication by project Chairman)
Solutions for Memory problem 1

1. Change the pipeline from spark room to ball valve for each spark.
2. Clean in the ball valve for each spark.
3. Drain additional gas.

Solutions for Memory problem 2

1. Change pipelines.
2. Clean in the spark room for each spark.
3. Additional gas.
5. Pre-operation of Shaving Sample

Etching using 6mol HCL  Sealed Bottle
Dry in the Clean Booth

Conclusion

1. Two different LBMA Au-RM were manufactured by the total technologies of Tanaka Kikinzoku group.

2. Homogeneity assay was done by both TKK by using Spark-Ablation-ICP and Rand Refinery Limited by using Spark-OES.

3. Homogeneity was assured by making the best use of ANOVA lead by the Steering Committee of the Project.

4. Contents of the RM have been assured by the 10 experienced major laboratories around the world by assaying the shaving sample of the RM raw material.

**LBMA RM-Project will strongly recommend you to procure the above Reference Materials for your Lab’s use.**
Thank you for your kind attention