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Slim-Fixed-Point-Cells

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METAL CLAD FIXED POINT CELLS FOR THE ITS-90

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Abstract

Traditionally primary laboratories have used quartz glass to clad graphite crucibles containing fixed point metals from the temperature scale [1].

For many years some laboratories such as National Research Council Canada (NRCC) have clad cells in metal because of its robust nature [2], [3], [4].

Isotech has produced metal clad fixed point cells from International Temperature Scale of 1990 (ITS-90) since 1990 [5], [6].

This paper presents the United Kingdom Accredited Service (UKAS) results from over 160 fixed point cells in quartz or metal clad plus 3 case histories which show that after proper preparation metal clad cells have similar performance to quartz clad cells.

Introduction

Does the metal cladding in metal clad fixed point cells contaminate the metal it clads?

Our experience is as a manufacturer of metal clad fixed point cells since 1990. The ITS-90 metals we clad are In, Sn, Zn and Al.

Larger, optimal cells have been quartz clad until 2007 with 1 or 2 exceptions. Smaller slim cells have been made to customers requirements in either quartz or more popularly in metal cladding.

If we take the slope of 70% to 80% of the melt or 50% of the freeze as a guide to purity we can use the quantity of cells we have made to statistically gauge any contamination from the cladding.

It should be noted that to test a cell it is melted and frozen three times and the melts and freezes are recorded. Thereafter the test cell is compared to a reference cell on at least two realizations of the freeze.

The purpose of these repetitions is precisely to note any slow changes in the cells performance due to for example contamination. We have never noted deterioration of the cells during calibration.

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Lot References

Over 18 years a large number of batches of each metal are represented, each has its own unique set of impurities.

In this short presentation it is not possible to reproduce all the certificates. A summary of the impurities is presented.

In selecting the metal to go into a cell the batches with the smallest impurities are selected for the larger more expensive cells.

The following tables (1 through to 4) compare the melt and freeze slopes of large and small cells together with the supplier's analysis of impurities; each table details a specific metal.

Table 1. Indium

Suppliers Analysis ppm	LOT Ref	50% Freeze mK	80% Melt mK	Number of Cells	Cell Size
0.3	6030	0.17	0.44	12	Large
0.3	1	0.17	0.44	12	Totals & Averages
0.6	990064	0.33	1.19	10	Slim
0.7	GS 1169	0.41	1.13	17	Slim
0.65	2	0.37	1.16	27	Totals & Averages

Table 2. Tin

Suppliers Analysis ppm	LOT Ref	50% Freeze mK	80% Melt mK	Number of Cells	Cell Size
0	2310	0.1	0.9	3	Large
0.3	7331	0.15	1.0	13	Large
0	23822	0.18	1.2	5	Large
0.6	8967	0.28	1.4	4	Large
0.3	4	0.18	1.1	25	Totals & Averages
0.3	7331	0.3	0.5	1	Slim
0.7	9387	0.45	1.67	8	Slim
0.6	8967	0.44	1.3	4	Slim
0	23822	0.23	0.97	3	Slim
0.75	1707	0.1	0.75	2	Slim
0.75	1707	0.43	1.25	9	Slim
0.61	6	0.325	1.1	27	Totals & Averages

Table 3. Zinc

Suppliers Analysis ppm	LOT Ref	50% Freeze mK	80% Melt mK	Number of Cells	Cell Size
0	7253	0.17	0.43	7	Large
0	7232	0.16	0.38	7	Large
0	7288	0.3	0.45	2	Large
0.5	11404	0.28	1.87	3	Large
0	9073	0.13	1.11	5	Large
0.1	5	0.21	0.84	24	Totals & Averages
1.0	2209	0.715	2.75	12	Slim
1.0	1	0.715	2.75	12	Totals & Averages

Table 4. Aluminium

Suppliers Analysis ppm	LOT Ref	50% Freeze mK	80% Melt mK	Number of Cells	Cell Size
0.2	4913	0.65	1.8	7	Large
0.5	9904	0.66	2.7	15	Large
0.35	2	0.655	2.2	22	Totals & Averages
0.5	9904	0.7	1.9	4	Slim
0.7	10068	0.9	3.2	11	Slim
0.9	1858	0.78	2.3	4	Slim
0.7	3	0.8	2.5	19	Totals & Averages

It is clear that the cells made from batches with better purity have less slopes, but there are no large differences that might be expected from contamination.

Case Study No. 1

The most difficult ITS metal to metal clad is aluminium because of its high temperature (660°C) and its sensitivity to contaminants.

During 2007 we obtained an ingot of very pure aluminium made for us by Pechiney of France.

We metal clad this material and compared the melts and freezes we obtained with our reference cell Al 188 which had been intercompared at National Institute of Standards & Technology (NIST).

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Table 5. Our Reference Cell (Al 188) is compared to a cell made of extremely pure aluminium

NRCC Anlaysis PPm	Lot Ref.	50% Freeze	70-80% Melt	Cell Type
0.9	Al 188 M9904	1.3mK	2mK	Quartz
0.3	Pechiney Al Al 6508	0.3mK	0.3mK	Metal Clad

The above results show clearly the superior performance of the Pechiney aluminium in its metal case Vs Al 188 in quartz.

Actual freeze and melt curves are presented (see Appendix 1).

It is clear that the metal cladding has not reduced the purity of the Pechiney aluminium.

Case Study No. 2

A large (or optimal) metal clad zinc cell serial number 192 was sent to NIST for intercomparison.

Three freezes and melts were performed on the cell, which showed no signs of deterioration during test. The cell was used in our UKAS primary laboratory until 2006 when it was replaced (UKAS require intercomparisons every four years, new cells are used because of the time taken to perform the intercomparison).

Case Study No. 3

A compete set of metal clad slim cells in a German DKD approved laboratory, complete with desk-top apparatus was sent to PTB for intercomparison in 2002 [7]

The results are tabulated below. The cells are still in continuous use!

Table 6. Summary of the intercomparison of Isotech slim cells with PTB National Standards

Slim Cell	ΔT mK	u/c mK
Hg 137	0	±1
Ga 123	-0.17	±0.25
In 125	-1.6	±2
Sn 132	+1.4	±2
Zn 64	+0.3	±3
Al 160	+1.0	±5

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Discussion

Cladding fixed points from ITS-90 in metal is not as simple as it seems. The metal used must be carefully prepared before assembly and carefully tested after assembly.

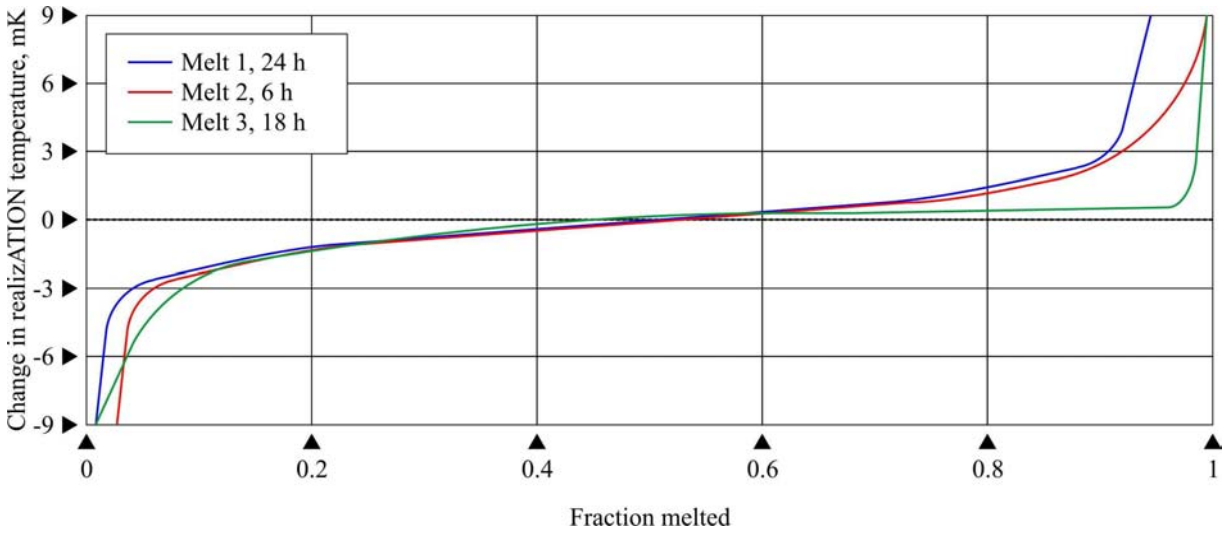
Subject to the above, evidence suggests that the metal cladding does not provide a source of contamination in either the short or long term.

The big advantage of metal cladding is robustness and transportability. Silver and copper have not yet successfully been metal clad.

Appendix

The following four graphs show actual freeze and melt curves as described in the text.

Graph 1. Melting curves for the Isotech Al fixed point cell (Isotech model ITL-M-17672-O, Serial No. Al 188



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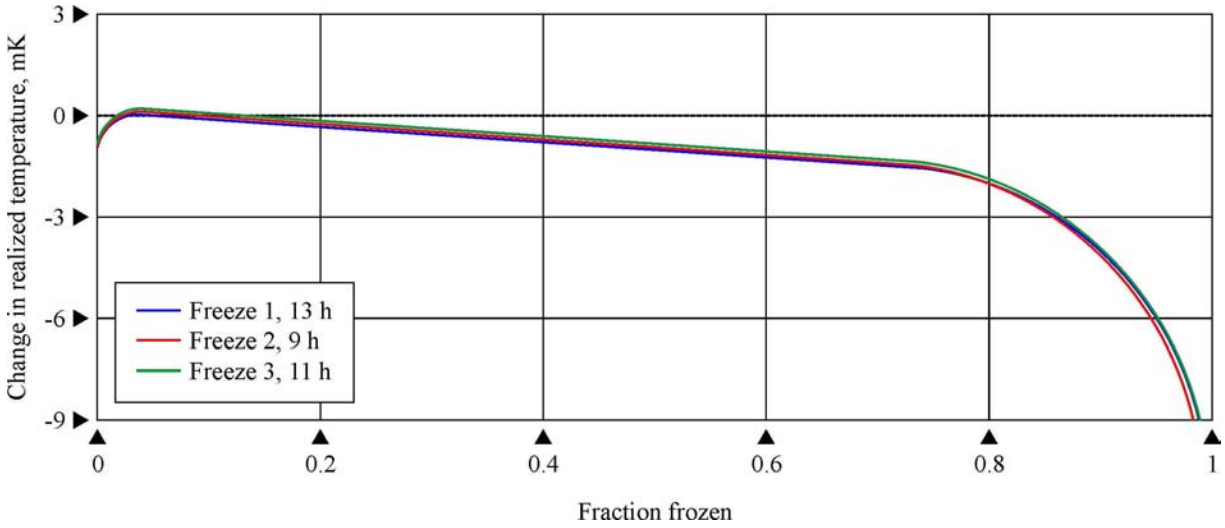
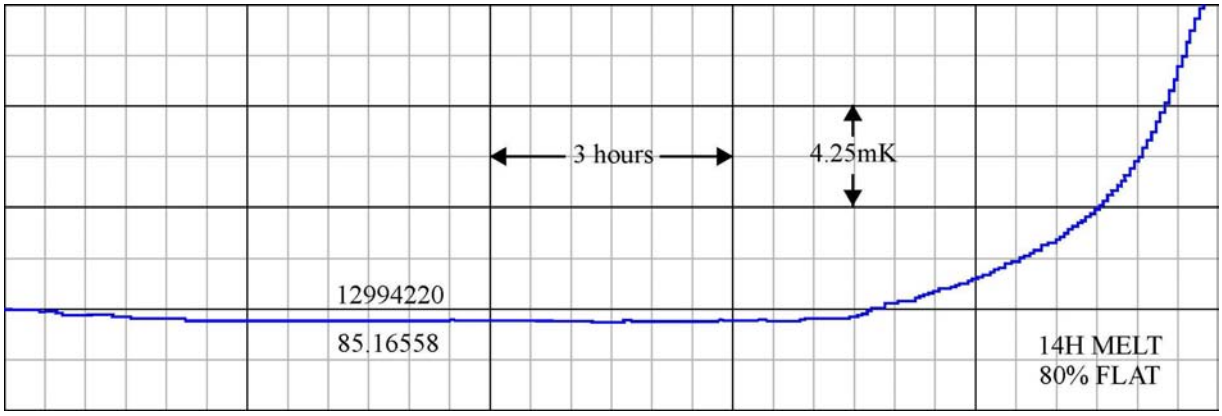
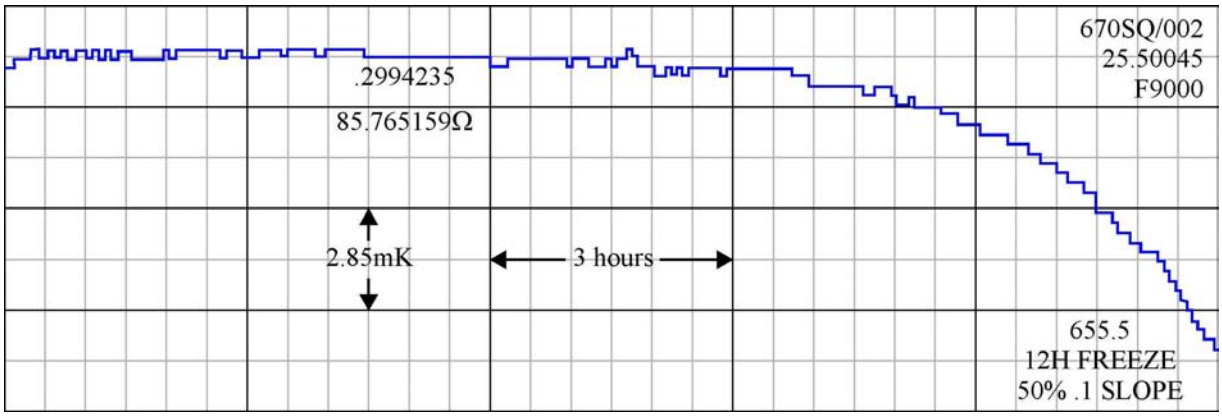
Graph 2. Freezing curves for the Isotech Al fixed point cell (Isotech model ITL-M-17672-O, Serial No. Al 188



Graph 3. Pechiney 6N5 Al melt curve



Graph 4. Pechiney 6N5 Al Freeze Curve



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