INTEC LTD

REVIEW OF CASTING BRIQUETTES TO COPPER ROD AT RAUTOMEAD, SCOTLAND AND WIRE DRAWING AT PHELPS DODGE, USA

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EXECUTIVE SUMMARY

Mr. P.J. Gabb, the principal of Mendip Metallurgy, Bristol, UK, attended casting trials at Rautomead International Ltd (RIL), Dundee, Scotland, on 11-12 November, 2003. The purpose of the trials was to evaluate the casting performance of Intec dendritic copper that had been produced during operation of a prototype electrowinning cell in Sydney, Australia, during July, 2003.

Dendritic copper from the electrowinning cell had been washed and dried before ram pressing into briquettes to facilitate transport to Scotland. A total of 1.8 tonnes of briquettes was available to cast into rod on a Rautomead vertical casting machine.

Rautomead Ltd is an accomplished manufacturer of horizontal and vertical continuous casting machines that are sold world-wide into the non-ferrous industries. Copper intended for wire drawing is usually first cast into rod using vertical machines. A typical cast rod diameter is 8mm as this facilitates the wire drawing operation. Accordingly, arrangements were made to cast the Intec material on a single head test machine capable of producing around 80kg/h of 8mm rod to ensure a reasonable period of continuous operation to identify any production upsets and material flaws.

Testwork commenced on Tuesday 11th November by melting a reservoir of around 450kg copper briquettes into a prepared carbon crucible. This operation identified an unexpected hygiene problem associated with melt fuming. The problem was overcome by relocating ventilation ducting to capture fume evolution above the point of briquette melting. A sample of fume was collected on a cold steel surface and was later identified as predominantly a copper phase with other contaminants not derived from copper dendrites.

Casting commenced and produced rod of excellent appearance until a point in the overnight operation. Around 250kg of rod was produced that contained voids, making it unsuitable for wire drawing. The problem was identified as probably being due to lack of graphite cover on the molten copper surface in the briquette melting region. Special attention was paid to ensuring an adequate graphite cover and good rod production resumed. The off-grade material was successfully remelted to make good product.

In the opinion of Mendip Metallurgy, the trial to produce copper rod from Intec briquetted dendrite copper has been successful. Although some fuming was experienced, this did not affect rod macro-physical quality over a period of some 20 hours of casting. Fuming is not anticipated to be a long-term problem and briquetted feed is not anticipated to be necessary in an integrated dendrite production and casting operation.

Subsequent to casting, the product copper rod was sent for drawing into fine wire at the facilities of Phelps Dodge in the USA. The Rautomead report of these trials is reviewed in this report with the conclusion that commercial quality fine wire down to 0.1mm diameter was produced from rod prepared from dendritic copper. Some residual uncertainty over wire oxygen content from the trials is not thought to be a major impediment to establishing a commercial operation.

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1 MAIN FINDINGS

Intec Ltd has developed a process for the hydrometallurgical extraction and recovery of base metals, more specifically copper, that it intends to commercialise with its Licensees in due course. The leaching, purification and electrowinning stages of the Intec Copper Process were previously piloted with various copper concentrates at a 1tpd of copper scale during 1998/99. Good metallurgical parameters were obtained. A new electrowinning cell has subsequently been prototyped using a full-size commercial electrode pair and has produced over 6t of dendritic copper. It has therefore been demonstrated that all hydrometallurgical stages of the process can be scaled-up to the commercial level at minimal technical risk.

In order to capitalise on the low costs of the Intec Copper Process, it is envisaged that high grade dendritic copper will be melted and cast into rod, then drawn to copper wire. In this way a high added-value commodity can be produced for sale on the world market. To establish these final physical metallurgical stages of the process it is necessary to trial the rod and drawing operations on representative quantities of ICP dendritic copper. To this end, 1.8t of dendritic copper from the full-size prototype electrowinning cell has been briquetted and transported to Rautomead International Ltd (RIL) in Dundee, Scotland, to conduct rod casting trials as a precursor to cold wire drawing at a further location. The trial is reviewed in this document to establish the technical, practical and operational suitability of Intec briquetted dendrites as feed to an upcasting vertical rod process.

The major findings of this review are:

- Briquetted copper dendrites from the Intec Copper Process provide a suitable feed for casting vertical rod utilising Rautomead equipment.

- High physical appearance 8mm diameter rod was produced that is believed suitable for direct drawing to fine copper wire.

- Some fuming and minor eruptions were experienced during briquette melting which is believed due to residual salts present in the dendrites. Improved washing technology will be utilised in commercial plant to overcome this minor issue.

- Copper tested in the trial originated from Grade A make-up to the electrowinning cell, not from copper derived from the leach and purification stages of the ICP. However, pilot plant testwork has demonstrated that Grade A purity copper is produced from Intec Copper Process leach solutions, so little technical risk is adopted in assuming equivalency of materials.

- Subsequent wire drawing trials conducted at Phelps-Dodge facilities in the USA have produced good quality fine wire down to 0.1mm diameter. It is anticipated that dendritic copper produced by the integrated Intec Copper Process will similarly be suitable for production of this high added value product.

2 CASTING EQUIPMENT

The casting of non-ferrous rod is either conducted horizontally or vertically depending on the nature of further processing. Horizontal casting is usually applied to produce larger diameter stock for processing to bar, tube, roundels, sheet, etc. Vertical casting is usually applied to produce rod typically between 6mm and 14mm diameter, mainly for drawing to wire for electrical cables. A common diameter for wire drawing is 8mm.

2.1 Rautomead Equipment

RIL manufacture a range of vertical casting machines that are referenced on the internet address www.rautomead.co.uk. The unique features are:

- Single melting and casting furnace for multi-strand casting
- Graphite crucible for high heat transfer and reducing environment
- Reliable, efficient, and safe, graphite resistance heating for good temperature control
- Quiescent metal bath with automatic cathode feed and level control
- Graphite flake cover with inert gas blanketing
- Semi-automatic operation with long die life
- Low annual downtime around 1-2 weeks
- Low production costs.

Commercial machines are usually equipped with crucibles large enough to accommodate multiple casting heads to achieve economic casting rates from single melters. Casting rate is determined by the rod diameter and the casting speed, the latter varying with the alloy being cast.

For test runs on small quantities, RIL use a single head RS80 machine rated at 80kVA, to give test periods in excess of 12 hours. A view of the test machine is shown on Figure 1. Graphite resistance radiant heating is supplied up to ratings of 800kg/h casting rate. Above this rating, induction heating is used with channel inductors. Outokumpu machines use a premelter and transfer the molten metal to the caster.

The casting operation is performed within an immersed graphite die set within a water-cooled insert. Copper freezes within the die and is withdrawn vertically in pulses of 3.7mm for good quality 8mm rod. A production rate of 80kg/hour/die is typical of commercial operation and requires a casting linear speed around 3m/min. This is achieved by setting a pulsing frequency of 13.5Hz in a variable frequency indexing gearbox linked to withdrawal rollers on the solidified rod above the die cooler. Rod is passed over a roller down to a coiling machine where it is pre-coated with Lubro 30 FM soap, as a 1% solution in water, to hinder surface tarnishing. The coiling operation is performed on standard equipment and is not considered a major variable in the test operation.

Figure 2.1 RS80 Casting Machine

2.2 Feed Material

Casting machines are usually set up to operate on a feed of standard copper cathode by submerging the cathode at a controlled rate according to the level of molten metal in the graphite crucible. In the case of small scale testwork, feed material is added manually at suitable periods to control the metal level within acceptable limits.

Melting dendrites into a quiescent metal bath is difficult due to their voluminous nature, low density and high surface area. The material does not sink readily into the molten metal but sits on the surface where it does not achieve high heat transfer rates for melting and is prone to oxidation, hence oxide slag production. However, around 80 tonnes of as-produced dendrites were previously melted in an induction furnace at the ICP pilot plant indicating that a turbulent surface, especially that produced in a channel induction furnace, overcomes melting difficulties.

As the RS80 casting machine relies on indirect radiant heating to a relatively quiescent bath, a decision was taken between Intec and Rautomead to compact the briquettes based on Rautomead success in handling and melting compacted turnings and swarf in alloy casting operations. Compaction was also favoured as it reduces dendrite oxidation in transit. Accordingly, dendrites were ram-pressed in Australia to produce 62mm diameter by typically 38mm long briquettes having densities between 92% and 95% of the theoretical 8.89 t/m^3. An illustration of briquetted dendrites awaiting feed to the casting crucible is given on Figure 2.2. The central division between melting and casting chambers is clearly visible on the figure.

Figure 2.2 Intec Briquetted Dendrites awaiting Feeding

Approximately 1.8t of this material was air-freighted to RIL for the casting trial, packed in 14-16kg lots in plastic bags, contained in large sealed plastic drums. The bags were purged with nitrogen before sealing to minimise copper oxidation en route but some surface oxidation was apparent. As-received briquettes are shown on Figure 2.3.

Figure 2.3 As-Received Briquettes

3 TEST RECORD

RIL has produced a comprehensive record of equipment, procedures and observations for the Intec trial, from which material is drawn for this section. Site notes were also recorded by Mendip Metallurgy to supplement the RIL record.

3.1 Crucible Conditioning

In the week preceding the Intec trial, the graphite crucible was conditioned by melting and casting Grade-A copper to wash out most of the impurities that might affect product quality. The crucible was allowed to soak for two hours at 1350C and an 8mm rod was cast for one hour to check the tooling, melting rate and coiling capabilities.

Rod samples were taken of the wash product against the need to check contaminants in the event that deleterious elements should be found in the Intec-sourced rod. The 450kg heel of copper left in the crucible after wash casting was drained out into a mould and the furnace allowed to cool down. Heating elements were checked and the crucible thoroughly cleaned out, then the assembly was rebuilt. No problems were noted.

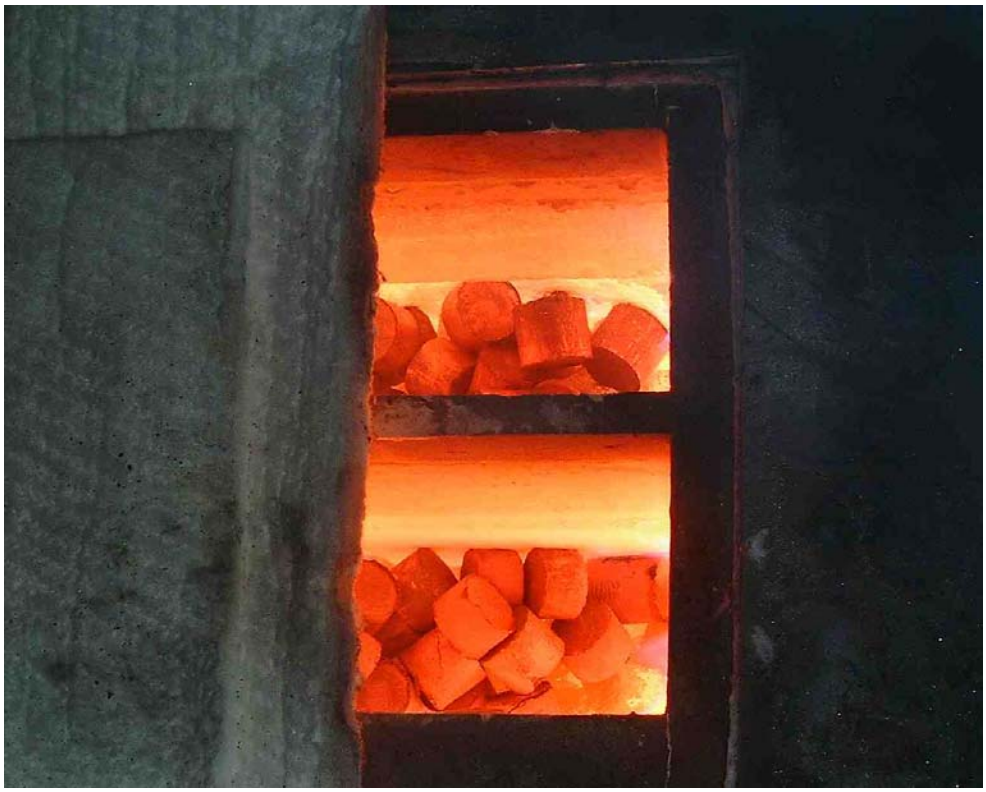
3.2 Briquette Melting

On Monday, 10 November, 2003, the crucible was preheated from cold using the graphite heaters to a holding temperature of 1,350C overnight. Standby power requirement was around 55kVA.

On Tuesday morning, a batch of 450kg of briquettes was weighed out and set ready for charging as the furnace heel. The furnace crucible has a dividing partition along its length that separates the melting and casting operations. An underflow weir between the two chambers allows melted metal to flow into the casting section thus providing time for superheating as it flows upwards to the casting die. The partition also provides the function of liberating any outgassing on melting prior to the metal flowing under the weir. Otherwise, de-gassing could affect the casting operation. The melting operation is underway in Figure 3.1.

It was immediately noticed that fuming occurred on adding the first briquettes to the preheated crucible. A ventilation duct was fitted to the side of the furnace top plate but proved insufficient to capture all fume, even with ceramic blanket laid across the chambers. The duct was repositioned above the melting chamber to more efficiently capture fume and briquettes were added to this side only by moving the blanket aside momentarily. Fuming is visible as a wispy light-coloured fume on Figure 2.2.

Figure 3.1 Briquette Melting in Progress



Fuming during initial heel melting operations had caused some disquiet with personnel within the shop. Fume had spread throughout the area causing nasal discomfort, and was thought characteristic of volatilised salts. The immediate view was that fuming was as a result of incomplete dendrite washing, giving a film of electrowinning cell electrolyte salts on the dried product. The only other supposition was that some ram press lubricant had been added during briquetting operations and that this was fuming off. The quantities of fume however were such that the former reason seemed most probable. A sample of fume was collected by condensing it onto a cold bar in front of the ventilation duct and taken to Intec for analysis.

The crucible was drossed-off on completing the addition of 450kg of briquettes, with evidence of some slag formation within the flake graphite cover on the melting chamber. A sample of this slag was taken, with the thought that residual oxide and chlorides may have been responsible. Spatter was also evident on the freeboard of the melting chamber walls. New graphite flake was added and the die assembly was inserted in the casting chamber. A stainless steel rod with a copper tip is inserted down the water cooled sheath into the graphite die. After a suitable time, the freedom of the copper tip to move up and down a small distance in the die was checked and casting commenced.

3.3 Casting on Day 1

Casting was ramped up automatically from 4% to 78% of full speed over a period of 20 minutes, the latter speed equivalent to 3m/min and 80kg/h. The stainless steel rod and residual copper stub is cut off as the copper rod emerges and the copper is routed over the idler roller to the coiling machine. Initially the rod had small surface voids but this problem cleared up after a few metres to give a smooth finish, typical of good quality rod.

Briquette charging was performed according to timed additions to equate to 80kg/h. Around half a plastic pack (around 7 briquettes) was added every 5 min against a stop watch. The briquettes were initially dropped in at the ventilation duct end of the melting chamber by pulling the ceramic blanket back momentarily. They were observed to mostly pass through the graphite flake layer, although some stayed visible for a period of a minute or so. As they assimilated into the melt a blue flame was produced and some minor eruptions occurred, but not so great that undue spatter was present. The blue flame seemed too intense to be associated with carbon monoxide combustion but no other cause was immediately apparent.

Minor surface defects again became apparent approximately 2h into the run. Melt temperature had gradually declined over this period from an initial 1,230C to the low end of the preferred range of 1,175C to 1,185C. It was suspected that some slag had formed in the die inlet and was impeding the flow of metal to the solidification zone. The cast was stopped and the cast rod was manually pushed back through the die by approximately 75mm to displace any build-up. Crucible metal level was also increased by around 25mm to give more residence time for slag separation in the melting chamber. Rod casting continued until 7pm on Tuesday under the parameters of Table 1.

Table 3.1 Casting Parameters

Main Casting Parameters				
Furnace Temp (C)	**Melt Temp Range (C)**	**Withdrawal Rate (m/min)**	**Water Inlet (C)**	**Water Outlet (C)**
1240	**1175-1185**	**3**	**30.7**	**34.7**

Water flow through the tool assembly was 50L/min to give a rod exit temperature less than 60C.

A power-down of the thyristor occurred at around 7p.m. that caused a temporary halt to casting when the crucible temperatures dropped. Power was restored and the melt reheated with casting recommencing around 90mins later when temperatures had been regained. It was notable during Tuesday that the power settings were fluctuating between 0% and 100% to control furnace temperature at 1240C. Whether this was associated with the power-down is unclear, but the PID control parameters were reset on Wednesday to give smoother power variations in the range 60% to 65% of full power to maintain the same furnace temperature.

At around 9p.m. rod surface quality deteriorated and rod exit temperature rose, indicating cooling problems. Casting was stopped and rod direction reversed to clear any build-up in the die. Casting restarted with acceptable quality but deteriorated to the point where the rod broke at 11:45 p.m. A new tool assembly was installed together with changing the graphite flake cover on the casting side, and casting restarted at 12:10 a.m. on Wednesday. Despite these changes the rod quality continued to deteriorate even though attempts were made to change the graphite melt covers on the casting side. The tooling was changed at 3:30 a.m. and a new die fitted, but rod quality remained bad until the rod again broke at 7a.m. when the furnace was put on standby.

3.4 Casting on Day 2

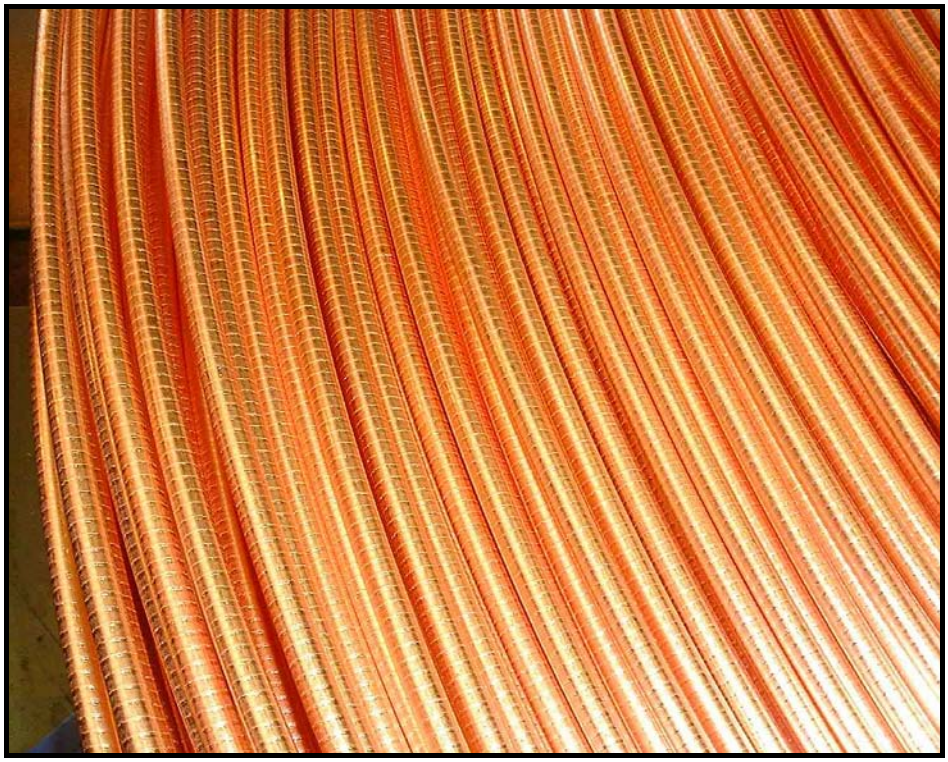
Only around 700kg of good rod had been cast on day 1 up to the point of the first rod breakage. A second coil of around 240kg of bad rod had been produced during the night shift which was not suitable for drawing to wire. Some 440kg of briquettes were left to convert.

It was thought that the problems of the night shift were not associated with briquette quality, as good rod was produced through Tuesday afternoon. RIL considered the problem lay with unstable melt caused by oxygen ingress through the melt surface. The casting chamber was clearly visible and was well covered with graphite flake, but the melting chamber had been covered with ceramic blanket to minimise fume emissions and was only pulled back part-way to add briquettes. The result was a gradual burning away of the flake graphite and exposure to the atmosphere. Spatter on the chamber walls indicated a “boiling” of the surface due to contact with oxygen and this was sampled for possible later analysis, if required.

A decision was taken to attempt to restore the melt to the condition on Tuesday and to restart casting. Both chambers were cleaned off and new graphite flake added. The ceramic blanket was left off the chambers so that surface condition could be easily monitored. The first graphite die that had been changed was sectioned along its length and observed to be virtually clean. There was a short length above the solidification zone where an unusual slight glossy zone was apparent, but this was not thought to be a cause of the difficulties. The tooling on the machine had only been used for 3.5 hours and was observed to be clean, so it was agreed to reuse it.

Casting recommenced at 11:05 a.m. utilising the same ramp-up settings. Rod quality was good from the outset and continued in the same condition until the last briquette was charged at 5:05 p.m. The scrap rod was then recharged to the crucible in an uninterrupted manner until 8:25 p.m. when all material had been converted into good quality rod. This run of 9h 20min (nominally 750kg) constantly produced high quality rod and confirmed that the good conditions of Tuesday afternoon could be replicated. An illustration of the good surface condition of coiled rod is shown on Figure 3.2.

Figure 3.2 Coiled Rod Surface Finish



4 MATERIALS TESTING

To some extent, the measurement of materials properties on the copper produced from the Intec prototype electrowinning cell is academic. The copper was derived from Grade-A scrap and dissolved in the cell anolyte to synthesise catholyte. The electrolyte itself was synthesised from essentially pure salts and kept in closed loop. Hence the normal potential contaminants such as sulphur, selenium, tellurium, bismuth, antimony, arsenic and lead are essentially absent, as confirmed by the dendritic copper assay during production on 21 July 2003:

Table 4.1 Dendritic Copper Assays

Element	Unit	Limit	19 July sample	21 July sample
Bi	ppm	<2	<0.1	<0.1
Se	ppm	<2	<0.1	<0.1
Te	ppm	<2	<0.1	0.2
Group	ppm	**<3**	**≤0.3**	**≤0.4**
As	ppm	<5	1.1	1.0
Cd	ppm		<0.1	<0.1
Cr	ppm		<0.1	<0.1
Mn	ppm		<0.1	0.2
P	ppm		<0.1	<0.1
Sb	ppm	<4	<0.1	<0.1
Group	ppm	**<15**	**≤1.6**	**≤1.6**
Pb	ppm	<5	0.3	0.2
S	ppm	<15	0.9	0.6
Co	ppm		<0.1	<0.1
Fe	ppm	<10	11.5	4.2
Ni	ppm		0.6	1.5
Sn	ppm		<0.1	<0.1
Si	ppm		<0.1	<0.1
Zn	ppm		<0.1	2.2
Group	ppm	**<20**	**≤12.5**	**≤8.2**
Ag	ppm	<25	16.8	12.6
TOTAL	ppm	**<65**	**≤32.4**	**≤23.6**

However, some of the largest effects of impurities in copper are produced by oxygen and hydrogen. Oxygen can be picked up by contact with air and hydrogen can be formed by contact with water vapour, such as could be present from inadequate drying of copper dendrites. So it is possible for these elements to have an impact on important properties such as tensile strength and elongation. Fairly high levels of contaminants can be tolerated in rod casting, but drawing rod to fine wire is very sensitive to residual contaminants that can induce cold shortness initially and then hot shortness with increasing contamination.

RIL took samples from the rod produced at the end of the "wash" run and from rod at the end of the two coils produced from briquettes. Samples from the coils were sent to Intec for chemical analysis at the same laboratory used to analyse the briquettes. Mechanical testing was performed at Abertay University, Scotland, with the results in Table 4.2:

Table 4.2 Mechanical Tests on 8mm Rod

Sample from:	UTS (N/mm^2)	% Elongation	% Reduction in Area
End of Wash Run	174	58.2	84
Start of Coil 2	183	60.7	87
End of Coil 2	192	60.0	88
EXPECTED	177	39-41	-

Coil 1 was not tested as it contains mixed quality rod. Electrical conductivity has not been measured as the RIL in-house spot test is not considered sufficiently accurate.

Phelps Dodge also performed chemical analysis on the incoming 8mm rod (Table 4.3), which can be compared with the results in Table 4.1. The chemical analysis was performed on a Thermo Jarrell Ash Atom Comp 2000 e-spectrometer at Phelps Dodge, Norwich, USA.

Table 4.3 Chemical Tests on 8mm Rod

Chemical Analysis (ppm)				Typical Rod made from Grade A ISA Cathode	
Coil No.1		Coil No.2			
Ag	11.0	Ag	11.7	Ag	9.3
As	n/d	As	n/d	As	0.86
Bi	n/d	Bi	n/d	Bi	0.24
Cd	n/d	Cd	n/d	Cd	0.05
Fe	1.3	Fe	1.3	Fe	0.7
Ni	n/d	Ni	n/d	Ni	0.19
Pb	n/d	Pb	n/d	Pb	0.28
Sb	n/d	Sb	n/d	Sb	n/d
Se	n/d	Se	n/d	Se	n/d
Sn	n/d	Sn	n/d	Sn	0.01
Te	n/d	Te	n/d	Te	0.05
S	4.0	S	3.5	S	8.0

Slag taken from the crucible surface after melting the 450kg heel was analysed in Australia, together with the fume sample taken during the melting operation. Results obtained are given in Table 4.4. The slag result is an average of two readings with close agreement.

Table 4.4 Slag and Fume Analysis

Sample	Units	Slag	Fume
Ag	ppm	2.8	21.5
Al	%	6.0	0.32
As	ppm	20	40
Bi	ppm	-	0.3
C	%	2.5	Insufficient sample
Ca	%	2.1	0.09
Cl	ppm	1100	Insufficient sample
Co	ppm	10	10
Cr	ppm	525	Insufficient sample
Cr (acid digestion)	ppm	-	515
Cu	%	35.5	45.2
Fe	%	2.4	1.73
Mg	%	0.44	0.02
Mn	ppm	545	265
Mo	ppm	277	1990
Na	%	1.21	1.19
Ni	ppm	60	-
Pb	ppm	10	60
S	%	0	0.02
Si	%	19.1	Insufficient sample
Ti	%	0.325	Insufficient sample
Ti (acid digestion)	ppm	-	70
W	ppm	79.5	884
Zn	ppm	197.5	590

Transverse photo micrographs of the 8mm rod were prepared and are compared with rod produced from cathode on Figure 4.1.

Figure 4.1 Rod Microstructure

It is seen that the rod produced from dendritic copper on the left has similar microstructure to that from cathode copper on the right. Longitudinal photo micrographs were not prepared.

5 WIRE DRAWING

Rod produced at RIL as two spools each of approximately 750kg was first transferred to Phelps Dodge Copper Products in Norwich, Connecticut, USA, where it was drawn from 8mm diameter to 1.38mm diameter. This product was then transferred to Phelps Dodge High Performance Conductors, Inman, South Carolina, USA, where it was further drawn down to 1.01mm. A report on both site trials during February, 2004, was produced by Rautomead International Ltd (Guy P Henderson) and is summarised in this section.

5.1 Drawing Trials at Phelps Dodge, Norwich

On receipt of the two coils of rod, the appearance of spool 1 was remarked as having many surface defects whereas spool 2 was thought of good appearance with no noticeable defects. Coil 1 material was produced during the initial trial period at Rautomead on November 11, 2003, when casting conditions were not particularly stable. Section 3.4 discusses the likelihood that problems were caused by oxygen ingress into copper in the melting side of the crucible. Improved melt cover with graphite and a deeper metal layer gave much improved conditions and led to the production of good rod on coil 2.

The Norwich facility does not have equipment to draw wire sizes smaller than 1.22mm, so Phelps Dodge drew the rod to 1.38mm on their rod breakdown machine on 5 February, 2004, prior to sending it to Inman for fine drawing. Coil 1 experienced an immediate break on drawing attributed to a casting defect. The break was retained for later examination, if required. A further 10kg was discarded from the coil and trials continued until approximately 200kg was drawn before another break, this time attributed to porosity. It was mentioned that the shape of a porosity break is similar to a tension break and can be incorrectly classified. In this situation the break could also have some dependence on the rod characteristics. The break was retained for examination. The remainder of Coil 1 was processed without further breakage.

Coil 2 rod was drawn to 1.38mm without problems. Drawing speed was 22m/s for the first 100kg increasing to the normal speed of 30m/s for the remainder. Elongation tests for the wire produced normal readings of 37.5% for Coil 1 material and 38.0% for Coil 2. Normal conductivity readings were measured at 101.4% IACS for Coil 1 and 101.3% for Coil 2. Low oxygen contents were measured at 1ppm for Coil 1 and 2ppm for Coil 2. Wire made from cathode copper would normally be expected to be in the 3-5ppm range, with the low level measured in test material possibly due to the efficiency of the graphite layer in removing oxygen in the melting crucible.

5.2 Drawing Trials at Phelps Dodge, Inman

The 1.38mm wire arrived by road at Inman on Monday 9 February. A SAMP TRB 4 drawing machine was used to draw the wire to 38 gauge (0.101mm). After one spool of wire had been produced a tension break occurred. The spool was changed for a larger size (DIN 250) and the drawing speed increased to 35.5m/s. Overnight at this setting a number of breaks were reported but no samples saved. The problem was adjudged to be with the annealing section of the machine, not the wire.

During operation on Tuesday, 10 February, at 35.5m/s, two breaks occurred in the annealing section whereupon the speed was reduced to 33m/s. A further break prompted removal of the dies for checking, and replacement of a guide pulley bearing in the annealer. Another two breaks occurred with a change in the wire wiper at the first break and rerouting of the wire in the annealer at the second. A further break prompted removal of the annealer from the wire path and the production of hard wire. The machine then ran uneventfully all night at 33m/s, indicating that the problems were indeed in the annealer. A typical wire elongation of 18-21% was obtained with the annealer in operation at normal setting.

On Wednesday, 11 February, speed was increased to 35m/s until all the wire from the bottom half of Coil 1 had been drawn. This material is presumed to come from the 8mm rod produced at RIL on the dayshift of February 11, 2003, which was observed by Mendip Metallurgy to be of good surface finish. The top half of Coil 1 was stated to have had multiple surface defects that would be unlikely to produce good wire, so upon reaching this material the machine was stopped and the remainder of the coil discarded. Mendip Metallurgy believes that this material could satisfactorily have been made into wire as most of the poor quality material was discarded at RIL and the coil was readily drawn to 1.38mm wire at Norwich after discarding some leading material.

Wire was drawn from Coil 2 during the remainder of Wednesday and all Thursday until 08.30 Friday morning. Two further breaks occurred on Wednesday of unknown cause and two occurred on Thursday nightshift attributed to machine problems, not the wire. Apparently only one hydrogen embrittlement test was performed on the wire, which failed. It was stated that the equivalent oxygen at failure would be 10ppm, in contradiction to the low measured values of 1 or 2ppm at Norwich. Accordingly, the wire could not be classified as “oxygen free – high conductivity (OFHC)”. The final physical appearance of 0.1mm diameter wire on a spool is shown in Figure 6.1.

Figure 5.1 Spooled Wire Appearance

6 RESIDUAL CONCERNS

Although the casting and wire drawing trials are considered to have been an overall success, there are issues that need to be addressed to ensure a trouble-free commercial process.

6.1 Clean Dendrite

The trials are believed to have identified problems either with the dendrite washing and drying operation of the prototype cell or with contamination in the briquetting operation, or with contamination in the melting crucible.

It is apparent from Table 4.1 that dendritic copper produced from the prototype cell was very clean, apart from a slightly elevated iron contamination in the first sample. However, by the time briquettes had been melted in the casting crucible, there were noticeably high levels of contaminants. Aluminium, calcium, iron, sodium and silicon accounted for up to one third of the slag weight. Copper accounted for just over one third, with the balance presumably mainly accounted by oxygen. Chloride levels were low, indicating a mainly oxidic slag.

Fume consisted primarily of copper but the colour was light, not the characteristic brown colour of copper oxide fume. Unfortunately there was insufficient sample for a chloride analysis to be performed, but the suggestion is that fuming elements were mainly associated with halogens. Some of the elements present in the slag and fume, namely iron, chromium, molybdenum and tungsten, indicate contamination from stainless steel. Other elements, namely silicon, titanium, magnesium and calcium, indicate contamination with sands.

It is not clear at this point how such contamination occurred. Chloride attack could have occurred in the washing and drying operation if this was carried out in stainless steel equipment. However, Intec has advised that stainless steel was only used in the final drying operation with nitrogen at low temperature and that no pitting corrosion is noticeable. All other washing equipment is made from titanium, hence no attack would have taken place.

Contamination with sand and steel particles could have occurred in the briquetting operation if the equipment was not fully cleaned before use. If briquetting is not a feature of commercial operation this contamination mechanism will be eliminated. Finally, contamination could have occurred from the casting crucible, but this mechanism is thought unlikely as the crucible was thoroughly prepared with a melt wash before melting dendrite briquettes. The analysis, given in Table 4.1, of dendrites straight from the electrowinning cell indicates very low levels of contamination of key elements, essentially removing the possibility that contamination was occurring during cell operation.

It was not possible to prepare a mass balance as the slag and fume could not be efficiently collected and weighed in the casting trials. However, the quantities of slag and fume were not massive, indicating a relatively small contamination that concentrated in these phases. There is a residual possibility that small levels of contaminants remaining in the cast copper

rod could influence drawing to fine wire. A recommendation is made to complete the rod chemical and micro-physical analyses, and compare these with the results of wire drawing.

6.2 Dendrite Charge

Most copper casting operations are geared to the use of cathode copper and have feed machines that lower the cathode into the melt at a controlled bath level. RIL has also successfully supplied melters for briquetted brass swarf with an automated feed system. It would be possible to provide such a system for briquetted copper dendrites but the additional expense of briquetting should be avoided if at all possible.

The problems with feeding dry dendrites to a copper bath are the low density of the material and its high surface area that encourages oxidation. There would also be the problem of traversing the flake graphite layer on the surface of the melt. As already described, the integrated Intec pilot plant melted 80 tonnes of dendrites in an induction melter and the resulting copper was sold. However, in a commercial operation, some forced feed system may be necessary to obtain a sufficiently high feed rate to meet the casting rate and to feed dendrites beneath the surface of the graphite covered melt.

Similar feed problems were experienced by the Compact Power process - see www.compactpower.co.uk that utilises tube pyrolysis to gasify industrial and domestic waste. The process is at the small commercial stage on a site near Bristol, UK, using a compacting screw feeder to feed waste into a ram press that pushes it through the pyrolysis tube. A similar system could be envisaged for feeding dendrites into a ram tube to push them under the surface of the melt. Voidage in the rammed dendrites would be greater than in briquettes and the contained gas would be liberated through the metal bath. Hence dendrites should be dried in an inert atmosphere to displace any oxygen, as currently envisaged in Intec plans.

Incorporation of dendritic material into the melter would be facilitated by the adoption of channel induction melting rather than radiant graphite heaters. RIL uses induction heating above copper melting rates of 800kg/h, and the introduction of compressed dendrites down a graphite tube above the channel up- or down-legs would enhance the melting process and avoid the need for a separate melter and caster. Care would need to be taken to ensure that any slag formation did not tend to build up on the walls of the channels.

6.3 Wire Embrittlement

The single hydrogen embrittlement tests performed on the final 0.1mm diameter wire failed, with the result that oxygen levels were projected to be above 10ppm. Although oxygen content is stated by RIL not to be a major parameter in preliminary casting and drawing trials, it is somewhat difficult to understand how the contamination occurred. Good rod production at RIL was accompanied by good graphite cover that would normally be expected to reduce oxygen content to very low levels, as apparently indicated by the Norwich tests.

If oxygen levels were indeed high, and incorrectly analysed at Norwich, it could be that metal circulation in the crucible at RIL was insufficient to provide good contact with the graphite. A channel inductor furnace would again be better for this purpose. Further analysis of this point

should be carried out by analysis of the remaining good wire material to determine whether there is a real problem with oxygen content. Mendip Metallurgy tends to agree with RIL in their statement that oxygen content is not a key indicator of quality in preliminary drawing trials such as those conducted at Phelps-Dodge.

RIL is convinced that breakages at Inman were associated with the equipment and not the wire. Primarily responsible was the annealer that apparently was only used for alloys, not copper. Furthermore, the drawing machine was a cone type machine with capstans close together that is more likely to produce tension breaks in the wire. If the wire had broken due to embrittlement the copper would break at the die, which was not the case at Inman.

7 CONCLUSIONS

Casting trials over the two-day period at RIL have provided further evidence of the suitability of copper from the Intec Process for processing to added-value products. Wire drawing trials at Phelps-Dodge facilities in the USA have indicated favourable performance in producing wire down to 0.1mm diameter. Conclusions for both trials are presented in the following sub-sections.

7.1 Casting Trials

- Briquetted dendritic copper from the Intec prototype electrowinning cell has been successfully converted to 8mm diameter copper rod as a precursor to further wire drawing operations.

- Some imperfections in rod quality occurred at the beginning and end of the first trial day that were overcome during the second day by ensuring the melt was adequately covered with flake graphite to prevent surface oxidation.

- Mechanical tests on the rod have produced excellent tensile and elongation results.

- Grain structure of the continuous cast 8.0mm rod from Intec dendrites showed similar characteristics to that of rod continuously cast from cathode.

- Briquette assimilation into the crucible melt was satisfactory with some evidence of fuming presumed to be caused by residual salts within the dendrites and possibly chloride attack on equipment. Commercial material is anticipated to have a very low salt content due to optimised dendrite washing and drying.

- Some slag formation was noticed on initial filling of the crucible, with an analysis that indicates a combination of some copper oxidation in the briquettes, surface oxidation of the melt due to lack of graphite cover, and contamination with extraneous material. No surface slag was noticed during casting, but some spatter on the wall was present due to minor eruptions during briquette addition.

- The second day trial duration of 9h 20min produced consistently good rod and was considered sufficient time to indicate any casting tool problems. The graphite die did not exhibit any signs of wear, blockage or oxidation.

- Normal commercial casting campaigns of up to seven days should be possible without interruption.

- Assimilation of dendritic copper into the melting crucible has previously been shown to be possible utilising a channel induction furnace. If either the rate of assimilation or oxidation tendency becomes problematic, a system similar to that used by the waste feeder into the Compact Power Ltd pyrolysis process could be successful. Briquetting should not be necessary in commercial operation.

7.2 Wire-drawing Trials

- Wire drawing trials conducted at Phelps-Dodge facilities in the USA have resulted in rod being drawn to fine wire at equal drawing speeds as good quality wire material currently used by PD in their production.

- A total of 384kg was drawn to 38 gauge (0.101mm) diameter fine wire out of a total cast rod total of some 1,500kg.

- Around half of the original rod was discarded either as a result of breakages during drawing or due to concerns over doubtful quality. Some 250kg of good quality 1.38mm wire remains.

- Wire breakages were adjudged to be mainly due to the wire annealer as the breaks essentially cleared when the annealer was taken out of circuit.

- The wire produced had normal electrical conductivity and elongation.

8 RECOMMENDATIONS

A number of recommendations are possible from the trial conclusions:

- Final chemical and micro-physical analyses should be completed on the copper rod and fine wire to establish any compositional differences that may indicate contamination.

- Fume extraction systems should be incorporated in any future design for dendritic copper melting to avoid any working atmosphere contamination, however small.

- Checks should be performed into the likely mechanism of the introduction of contamination into dendrite briquettes to ensure fuming and slag formation are minimised in commercial operation.

- Doubts over the embrittlement and oxygen content of the final 0.1mm diameter wire should be resolved by further metallurgical testing and chemical analysis.