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EXOTHERMIC AND RESISTANCE STARTING TECHNIQUES FOR USE IN THE EL--ETC(U)
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MELBOURNE, VICTORIA

REPORT

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EXOTHERMIC AND RESISTANCE STARTING TECHNIQUES
FOR USE IN THE ELECTROSLAG-REFINING PROCESS

Richard C. Andrew, George M. Weston,
John R. Bentley and Lance D. Redman

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ABSTRACT



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Two cold-starting devices based on exothermic reactions and resistance-heating, respectively, have both been developed to the stage where reliable starting can be achieved. A common feature of these devices is that they are readily attached to the end of the consumable electrode. Although the exothermic device produced faster starts, some contamination of the ingot resulted from its use. The exothermic device also resulted in the formation of worm-hole porosity in the first 25 mm of ingot length.

Ingots produced with the resistance device were free from both these forms of contamination and, therefore, the use of this device is preferred for the experimental work at MRL.



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Two cold-starting devices based on exothermic reactions and resistance-heating, respectively, have both been developed to the stage where reliable starting can be achieved. A common feature of these devices is that they are readily attached to the end of the consumable electrode. Although the exothermic device produced faster starts, some contamination of the ingot resulted from its use. The exothermic device also resulted in the formation of worm-hole porosity in the first 25 mm of ingot length.

Ingot produced with the resistance device were free from both these forms of contamination and, therefore, the use of this device is preferred for the experimental work at MRL.

DEFENCE RELEVANCE STATEMENT

The electroslag-refining process is a relatively new technique for the production of premium-quality alloys for critical applications such as ordnance. MRL is currently examining the potential of the process for the production of materials of interest to the Defence Services, particularly gun steels, under Task Number DST 77/062 and the present investigation forms part of this task.

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EXOTHERMIC AND RESISTANCE STARTING TECHNIQUES FOR USE
IN THE ELECTROSLAG-REFINING PROCESS

1. INTRODUCTION

A major problem confronting the operator of an electroslag-refining (ESR) furnace is to obtain a consistent and reliable starting procedure. This problem is compounded by the need to ensure that the starting procedure is not detrimental to ingot yield, i.e., the surface finish, soundness and structure of the ingot adjacent to the base-plate should be comparable, as far as is practicable, with that of the remaining ingot.

Starting difficulties are directly attributable to the unique method of heat generation of the ESR process and to the types of slag used. This process relies upon the resistance heating of a molten slag bath to generate sufficient heat to melt the consumable electrode [1]. This electrode is immersed in the slag bath and forms one pole of the electrical circuit. Thus, the creation of the molten slag bath is an essential first step in the remelting operation. The slag is generally composed of calcium fluoride with additions of lime and alumina, and is only electrically conducting when molten. For this reason, the powdered slag cannot be simply added to the mould and power then applied to obtain a start.

The starting techniques which have been developed fall into two categories, namely, liquid starts and cold starts. To obtain a liquid start, the slag components are fused in a special slag furnace which may be attached to the ESR furnace. The fused slag is then transferred to the ESR mould and power applied to the consumable electrode. This technique requires expensive auxiliary equipment and its use is generally restricted to large diameter moulds, as rapid solidification of the fused slag can occur in small diameter moulds (< 200 mm in dia.) before a start is achieved. The ESR moulds in use at MRL fall into this latter category.

To obtain a cold-slag start, the powdered slag is added directly to the ESR mould. Sufficient heat is generated (see later) *in situ* to fuse a small portion of the slag, and this then conducts current between the electrode and the base-plate. Eventually the whole slag charge is fused. Several heat sources have been employed for the initial slag fusion, the most popular being to arc between the electrode and the base-plate. However,

experience at MRL and elsewhere [2] has shown that this method of heat generation is unreliable and difficult to control. For example, powdered slag encroaching between the electrode and the base-plate can extinguish the arc, while welding of the electrode to the base-plate can occur under certain electrical conditions [3]. Furthermore, ingot quality adjacent to the base-plate is often unsatisfactory.

Other cold-start techniques which have been investigated include the use of starting tablets which are placed between electrode and base-plate. Some of these tablets are exothermic in nature [4], while others contain an electrically-conductive slag component such as a modified form of titanium dioxide [5]. However, little information is available on the composition of these tablets, their effectiveness, and on the ingot contamination that results from their use.

Consideration has also been given to the fusion of slag *in situ* by resistance heating of the slag using non-consumable resistance elements attached to the end of the electrode. However, this method suffers from one major drawback when used in long, static moulds. Once the slag is fused, the electrode must be withdrawn from the mould, the non-consumable elements removed, and then the electrode reimmersed in the slag. During this time the slag may solidify, thereby preventing a restart.

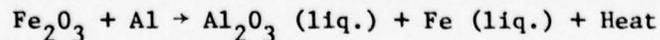
Accordingly, cold-start techniques for the production of small stable slag pools by either exothermic or resistance heating of the powdered slag have been examined at MRL. Both techniques have been developed to the stage where reliable starting can be achieved.

2. DEVELOPMENT OF AN EXOTHERMIC STARTING COMPACT

2.1 Exothermic Materials

The selection of the exothermic materials for investigation is governed by a number of factors. For instance, a suitable exothermic material must safely and reliably provide sufficient energy to melt the minimum amount of slag required to ensure electrical contact between the electrode and the base-plate without affecting the purity and quality of the ingot. Other factors include the ease of ignition of the exothermic reaction, availability, cost, and ease of handling. The family of materials known as thermites best meet these requirements. Thermites consist of a metal fuel and a metal oxide oxidant.

In a typical thermite the reaction proceeds as follows :



The heat generated by the reaction will raise the temperature of the reaction products to a level which is dependent upon, amongst other things, the specific heats and latent heats of phase changes of the products. For most thermites the temperature is limited by the boiling or sublimation of one

of the reaction products; in the case quoted above the temperature is limited by the boiling of iron at about 3000°K. Gas is not a product of the exothermic reaction, but some gas may be evolved due to the boiling of a reaction product, the presence of voids in the pressed composition, small amounts of hydrogen absorbed onto the surface of the metallic fuel and organic impurities.

Iron oxide/aluminium was the first system studied, the reason being that the reaction products, iron and alumina, are normally present in the furnace. Titanium, magnesium and silicon were later included as fuels in order to vary the ignition characteristics and burning rate of the thermite, and the physical properties of the reaction products. The mixtures tested, and the calculated heat output for each individual fuel/oxidant reaction, are given in Table 1.

Initial tests were carried out on 25 mm diameter compacts, pressed at 35.5 MPa, which contained a 6 mm diameter steel spigot (see Fig. 1(a)). The inclusion of this spigot enabled the exothermic reaction to be initiated by resistance heating. Two arrangements of the compacts were tried, as shown in Fig. 1(b) and (c). Early in these tests it was found that, once the thermite had been ignited, electrical contact between the electrode and the base-plate could not be maintained because unfused slag between the flat end of the electrode and the base-plate prevented the electrode from moving closer to the base-plate. To overcome this problem, a conical end was machined on the electrode for subsequent tests and this produced promising starts. However, several other problems were then encountered. Firstly, after ignition the compact burnt rapidly and quickly collapsed, necessitating undesirably large, rapid downward movement of the electrode to regain electrical contact. Furthermore, gases evolved during the exothermic reaction forced surrounding slag away from the tip of the electrode rather than allowing it to fill the void between electrode and base-plate. It was also difficult and time-consuming to shape the electrode end and to position the compacts on the base-plate such that contact between the end of the spigot and the electrode could be made without damaging the compact.

2.2 Attachable Compacts

An attachable compact was designed to overcome these problems. Important features of this design are :

(a) *The size and shape of the steel core.*

- (i) The small diameter spigot on the end of the steel core was kept as short as practicable (see Fig. 2). This minimised the downward movement of the electrode required to maintain electrical contact once the thermite ignited.
- (ii) The dimensions of the spigot were selected such that the current required to heat the thermite to its ignition temperature (approximately 900°K) was similar to the operating current of the furnace, thus overcoming the need for the operator to make large adjustments to the current when a start was achieved.

- (iii) The conical shape of the body of the steel core enabled electrical contact to be quickly and easily regained once the spigot melted.
- (b) *The distribution of the thermite around the steel core.*
- (i) Pure thermite has been retained around the spigot to facilitate ignition, but the bulk of the thermite has been replaced with a 60% thermite/40% slag blend (see Fig. 3(a) and Table 2). The addition of slag to the thermite reduces both the burning rate and the temperature of the reaction products. Calculations indicate that the reaction temperature is reduced from 3000 to 2600°K (see Appendix I). The reduced burning rate improves the ease of starting by allowing the operator more time to manipulate the electrode to follow the exothermic reaction. Furthermore, at the lower reaction temperature none of the reaction products will boil. This reduces the quantity of evolved gases, and thus allows the slag to fill the void created by the exothermic reaction. The composition of the various thermite/slag blends is listed in Table 2. The slag composition was not found to be critical and may be a pure component such as CaF_2 or a slag mixture. In the MRL work, pure CaF_2 was blended with the thermite to avoid contamination of the bulk slag. Satisfactory burning rates were achieved with a 60% thermite/40% CaF_2 blend.
- (ii) The third layer, which consisted entirely of slag, provides additional material to fill the void created by the exothermic reaction. Again the composition of this layer is not critical, and the use of a slag compatible with the bulk slag composition is recommended. Different masses of the three layers were studied, optimum starting performance being achieved with the masses given in Table 2. The exothermic materials and slag are enclosed in thin aluminium foil as shown in Fig. 3(b).

To obtain a start the compact is attached to the end of the electrode and the spigot is brought into contact with the base-plate. The required quantity of powdered slag is then placed around the compact and the end of the electrode. Sufficient current is passed through the spigot to initiate the exothermic reaction.

2.3 Performance of the Attachable Compacts

A series of 70 mm diameter steel ingots was produced using attachable compacts containing the exothermic materials listed in Table 2 to initiate melting. The bulk slag composition was 70% CaF_2 /30% CaO by weight. Excellent, easily-controlled starts were obtained with all four thermite compositions using both the d.c. electrode positive and d.c. electrode negative power modes. The current and voltage traces in Fig. 4 reflect the ease with which a smooth transition from compact ignition to stable melting conditions was attained. The compacts containing titanium were more difficult to ignite by resistance heating of the spigot than the other compacts,

and a small amount of arcing was required to initiate the exothermic reaction. This was achieved by applying power to the electrode and then slowly withdrawing it from the base-plate. Furthermore, the titanium compacts were the slowest to establish sufficient fused slag to enable the commencement of stable melting. This was attributed to the relatively low heat output of the titanium/iron oxide reaction (Table 1).

Popping noise nearly always accompanied compact ignition. The source of this noise was not specifically determined but it may be due to out-gassing of the compact or localised boiling during the exothermic reaction, or to the presence of moisture in the slag. Much less noise was produced by the titanium compacts and, as the heat output of the titanium/iron oxide reaction is lower than that of the other reactions, this suggests that localised boiling is largely responsible.

Worm-hole porosity was detected in all ingots and, in all cases, was confined to the first 25 mm of ingot length as shown in Fig. 5. Apart from porosity, ingot quality adjacent to the base-plate was comparable with the rest of the ingot length. Chemical analyses surveys revealed that the use of the attachable starting compacts caused some contamination of the ingots with either aluminium, aluminium and magnesium, silicon and magnesium, or titanium, depending on the thermite composition. This contamination, which was generally more pronounced in the ingots made with the d.c. electrode positive power mode, was highest at the ingot base, and decreased to normal ingot concentrations further along the ingot length (see Fig. 6). Silicon fell below the normal ingot concentration in the first 50 mm of the ingot length, while aluminium, magnesium and titanium persisted at higher than normal concentrations for a considerably greater distance along the ingot length. The question of whether or not this contamination is detrimental to ingot quality is beyond the scope of the present investigation. However, if the attachable exothermic compacts are to be scaled up for commercial ingot production, contamination is one problem which would need to be considered. Two factors which should be taken into account are the specification for the ingot composition, and the relative weights of the thermite and the ingot.

3. COLD STARTS USING RESISTANCE HEATING

3.1 Development

Preliminary studies were conducted using the arrangement shown in Fig. 7(a). The resistance element was a cylindrical steel spigot which was welded to the base-plate. The electrode was brought into contact with the top of the spigot to complete the electrical circuit, and powdered slag was placed around the spigot and the end of the electrode. The use of a consumable resistance element overcame the need to withdraw it from the mould once slag fusion had occurred. Contamination of the ingot by the consumable spigot can be avoided by making the spigot from the same material as the electrode.

To obtain a start, the temperature of the spigot is gradually raised to the slag melting point by increasing the amount of current passing through the spigot. Although slag in contact with the spigot is fused by this procedure, a start has not yet been achieved because the spigot is still solid and continues to carry the bulk of the current. The current is raised further to melt the spigot and, in the process, more slag is fused. By the time the spigot melts, sufficient fused slag has been generated to ensure a start, provided that electrical contact can be maintained by driving the electrode closer to the base-plate; hence the need for a conical end on the electrode.

Although controllable starts were obtained with this procedure, it was not entirely satisfactory for a number of reasons. For instance, it was difficult to align the electrode and the spigot when the mould was in position. Further, the machining of the conical end on the electrode was awkward and time-consuming. Also, at times, it was not possible to regain electrical contact with the fused slag after the spigot melted.

These problems were overcome by attaching the steel core of the above-mentioned exothermic compact to the electrode to provide the conical end-piece and using its spigot as the resistance element.^{7,7c} It was found that starting reliability was affected by the length of the spigot, optimum results being obtained when this was increased from 8.5 to 13 mm. It was also found that starting reliability could be improved considerably by preheating the slag for several minutes before the current was raised sufficiently to melt the spigot. Recommended preheating current ranges for mild steel spigots of various diameters are shown in Fig. 8, together with the currents necessary to melt these spigots.

3.2 Results

Several 70 mm diameter steel ingots were made using the attachable resistance element to initiate slag fusion. In all cases, the slags were well preheated before the current was increased to melt the spigot. Figure 9 shows the ease with which stable melting conditions were achieved following the use of such a preheating schedule. Excellent, easily-controlled starts were obtained on all occasions, the average starting time, including the preheating stage, being approximately 10 minutes. The resistance starts were much quieter than the exothermic starts, and this is attributed to the greater control which the operator has over the heat input. Ingot quality adjacent to the base-plate was equal to the rest of the ingot length and, most importantly, this region of each ingot was porosity-free.

4. SUMMARY

1. Two cold-starting devices based on exothermic reactions and resistance-heating, respectively, have been developed for use in the ESR furnace at MRL.
2. Four exothermic reactions have been examined and each produced satisfactory slag fusion.

3. A common feature of the exothermic and resistance devices is that they can readily be attached to the end of the consumable electrode, thus considerably reducing the time and difficulties involved in the pre-melt preparation.
4. Both devices are consumed during slag fusion, the exothermic compact causing some contamination of the ingot length, whereas ingot contamination arising from use of the resistance device can be completely eliminated by suitable selection of material for the resistance element.
5. While reliable, easily-controlled starts were obtained with both devices, the total starting time for the exothermic compacts was approximately half that required for the resistance starts. The resistance starts were, however, considerably quieter than the exothermic starts.
6. Use of the exothermic starting compacts resulted in the formation of worm-hole porosity in the first 25 mm of ingot length.
7. The resistance device is preferred for the experimental work at MRL because of the resultant higher ingot quality. The feasibility of scaling-up the device for the production of larger ingots needs to be assessed.

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T A B L E 1

EXOTHERMIC MIXTURES SELECTED FOR ASSESSMENT

Basic Thermite System	Thermite Mixture Selected	Metal Oxide Oxidant, Fe ₂ O ₃	
		Fuel	Energy Released, kJ/g of Reactants
Fe ₂ O ₃ /Al	Fe ₂ O ₃ /Al	Al	4.02
Fe ₂ O ₃ /Mg	-	Mg	4.60
	Fe ₂ O ₃ /Mg + Al	Mg + Al	$\frac{4.60 + 4.02}{2} \approx 4.3$
Fe ₂ O ₃ /Si	-	Si	2.38
	Fe ₂ O ₃ /Si + Mg	Si + Mg	$\frac{2.38 + 4.60}{2} \approx 3.5$
Fe ₂ O ₃ /Ti	Fe ₂ O ₃ /Ti	Ti	2.55

T A B L E 2

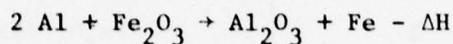
COMPOSITION OF THE ATTACHABLE EXOTHERMIC COMPACTS

Attachable Compact	Zone (see Fig. 3(a))	Constituents in each Zone (mass of each constituent, g)
Fe ₂ O ₃ /Al	1	Fe ₂ O ₃ (11)/Al (4)
	2	Fe ₂ O ₃ (16)/Al (8)/CaF ₂ (6)
	3	CaF ₂ (10)
Fe ₂ O ₃ /Al + Mg	1	Fe ₂ O ₃ (11)/Al (2) + Mg (2)
	2	Fe ₂ O ₃ (16)/Al (4) + Mg (4)/CaF ₂ (6)
	3	CaF ₂ (10)
Fe ₂ O ₃ /Ti	1	Fe ₂ O ₃ (14)/Ti (6)
	2	Fe ₂ O ₃ (15)/Ti (9)/CaF ₂ (6)
	3	CaF ₂ (10)
Fe ₂ O ₃ /Mg + Si	1	Fe ₂ O ₃ (11)/Mg (2) + Si (2)
	2	Fe ₂ O ₃ (16)/Mg (4) + Si (4)/CaF ₂ (6)
	3	CaF ₂ (10)

APPENDIX I

TEMPERATURE OF REACTION OF THERMITES

(1) Aluminium/iron oxide (Stoichiometric)



$$\Delta H = (-1690 + 837) \text{ kJ}$$

$$= -853 \text{ kJ}$$

Heat required to heat products to 3000°K

(a) 2 mol of iron	245 kJ
-------------------	--------

(b) 1 mol of alumina	427 kJ
----------------------	--------

	<hr/>
	672 kJ

Residual heat, ≈ 180 kJ, is sufficient to boil some of the reactants.

(2) Aluminium/iron oxide/calcium fluoride (26%/54%/20% by wt.)



$$\Delta H = -853 \text{ kJ}$$

Heat required to heat products to 2600°K

(a) 2 mol of iron (liquid)	203 kJ
----------------------------	--------

(b) 1 mol of alumina (liquid)	372 kJ
-------------------------------	--------

(c) 1.1 mol of calcium fluoride (liquid)	272 kJ
--	--------

	<hr/>
	847 kJ

Thus temperature of reaction is about 2600°K

Values of thermodynamic data used are given in the accompanying table.

APPENDIX I

(Continued)

Substance	+Heat of formation, kJ/g mol	+Heat of transition, kJ/g mol	+Temperature of Transition °K	Empirical Heat Capacity Values,* J/g mol		
				a	b	d
Al ₂ O ₃ (s)	- 1690	108.0	2300	109.3	18.37	- 30.42
Al ₂ O ₃ (l)			decomposes	138.1		
Fe ₂ O ₃ (s)	- 837					
Fe (s), α		1.67	1033	14.1	29.7	1.80
Fe (s), β		0.84	1180	43.5	-	-
Fe (s), γ		0.63	1673	20.21	12.6	-
Fe (s), δ		16.2	1808	43.1	-	-
Fe (l)		354	3008	4.18	-	-
CaF ₂ (s)		0.004	1424	59.62	30.33	1.97
CaF ₂ (s)		29.6	1691	107.57	10.42	-
CaF ₂ (l)			2773	99.6	-	-
SiO ₂ (s)	- 868					
TiO ₂ (s)	- 956					
MgO (s)	- 756					

+ Values from references (6) and (7)

* Empirical heat capacity equation.

$$C_p = a + (b \times 10^{-3}) T + \frac{d \times 10^5}{T^2} \quad (\text{ref. 6})$$

$$H_t - H_{298} = \int_{298}^t C_p dT$$

Where T is the absolute temperature.

a, b, and d are determined empirically, their values are given in tabular form in references (6) and (7).

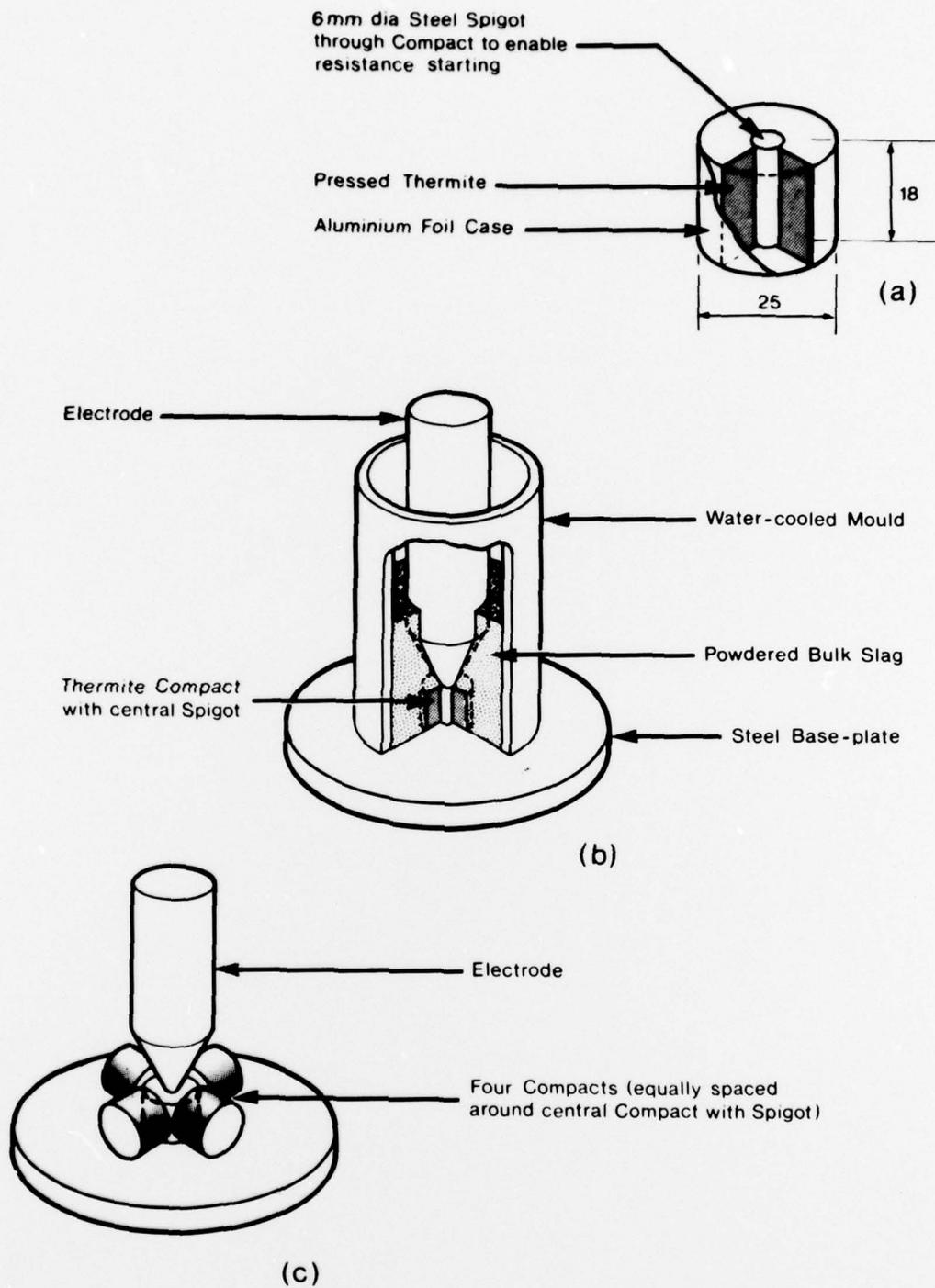


FIG. 1 - Shape and location of the 25 mm dia. exothermic compacts.
 (a) Compact dimensions.
 (b) Position of compact in the mould.
 (c) Arrangement of several compacts for increased heat output.

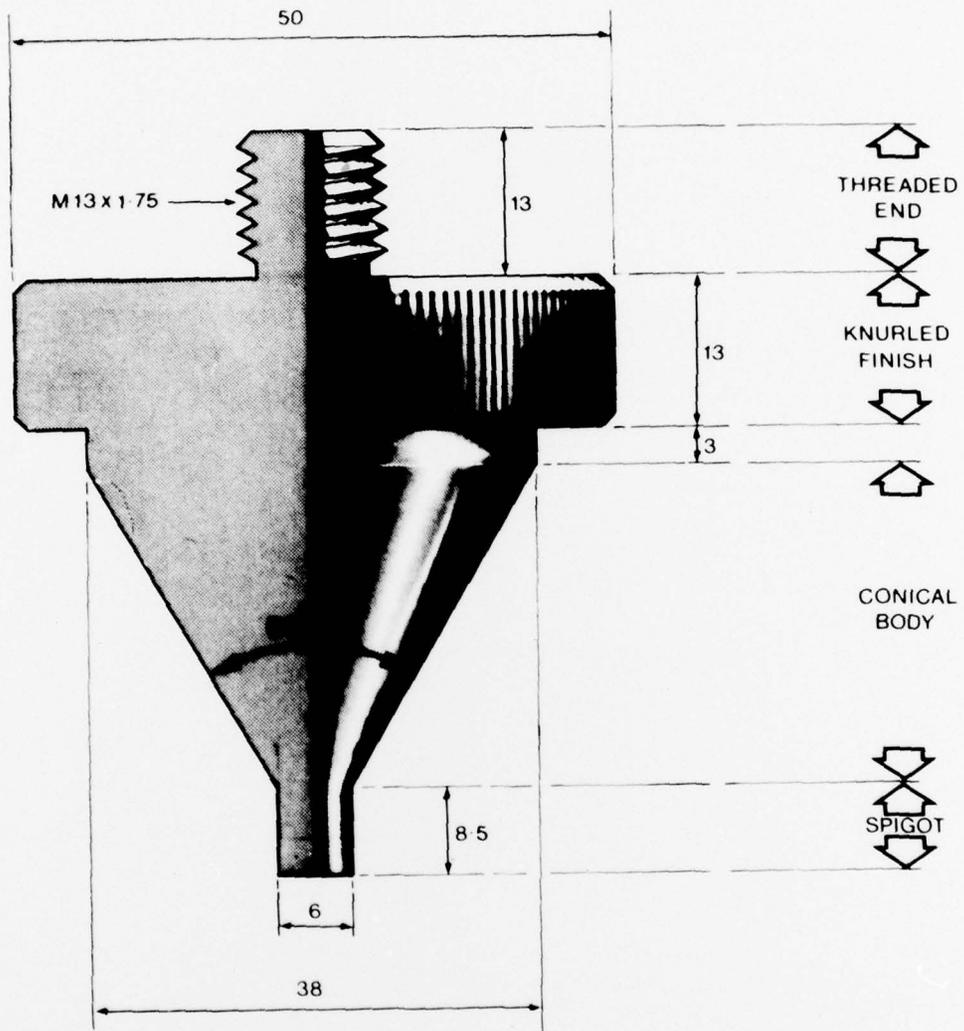


FIG. 2 - Size and shape of the steel core used in the attachable exothermic compacts.

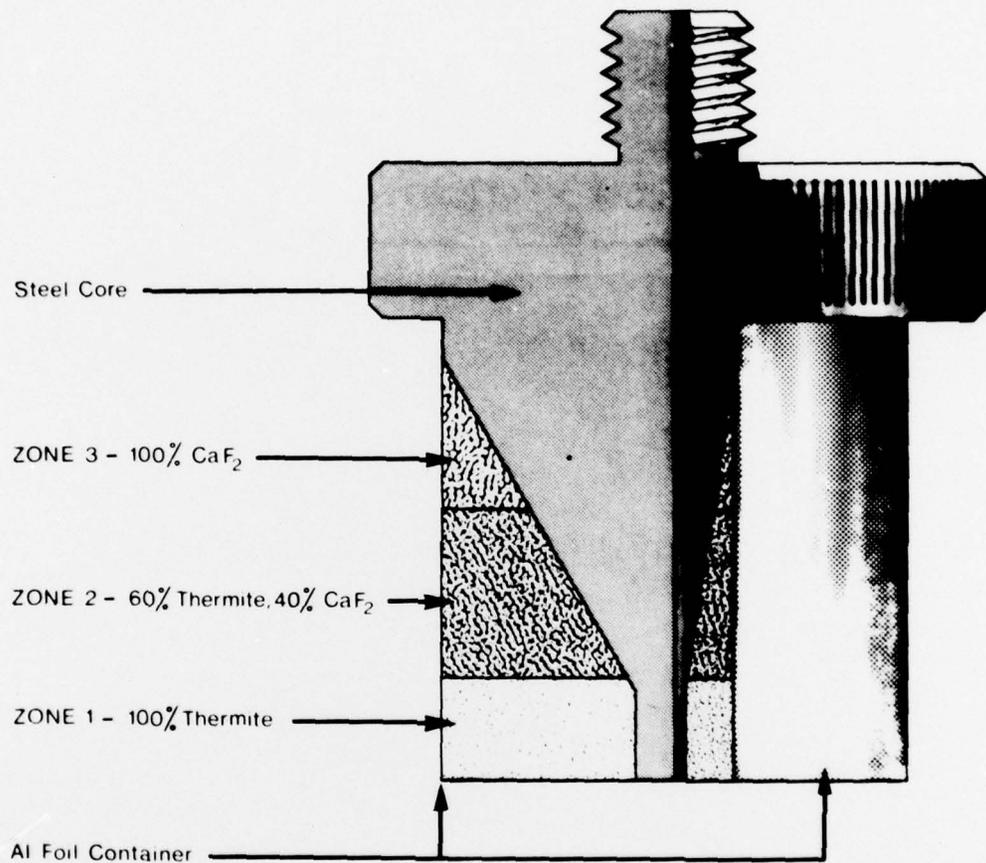


FIG. 3(a) - Section through an attachable exothermic compact showing the distribution of the thermite and slag.



FIG. 3(b) - External appearance of the exothermic compact.

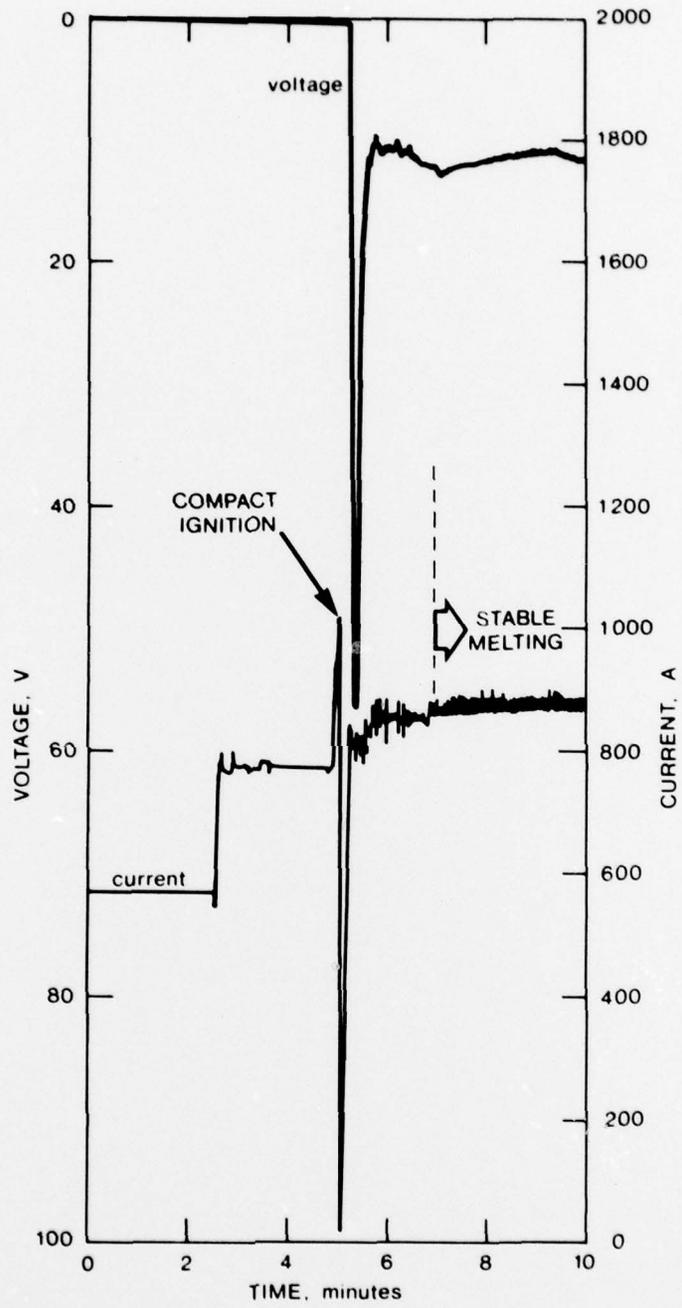


FIG. 4 - Voltage and current traces for an exothermic start (Al/Fe₂O₃ compact).



FIG. 5 - Section through the centre-line of a 70 mm diameter ingot formed with an attachable exothermic starting-compact. Porosity is confined to the first 25 mm of ingot length.

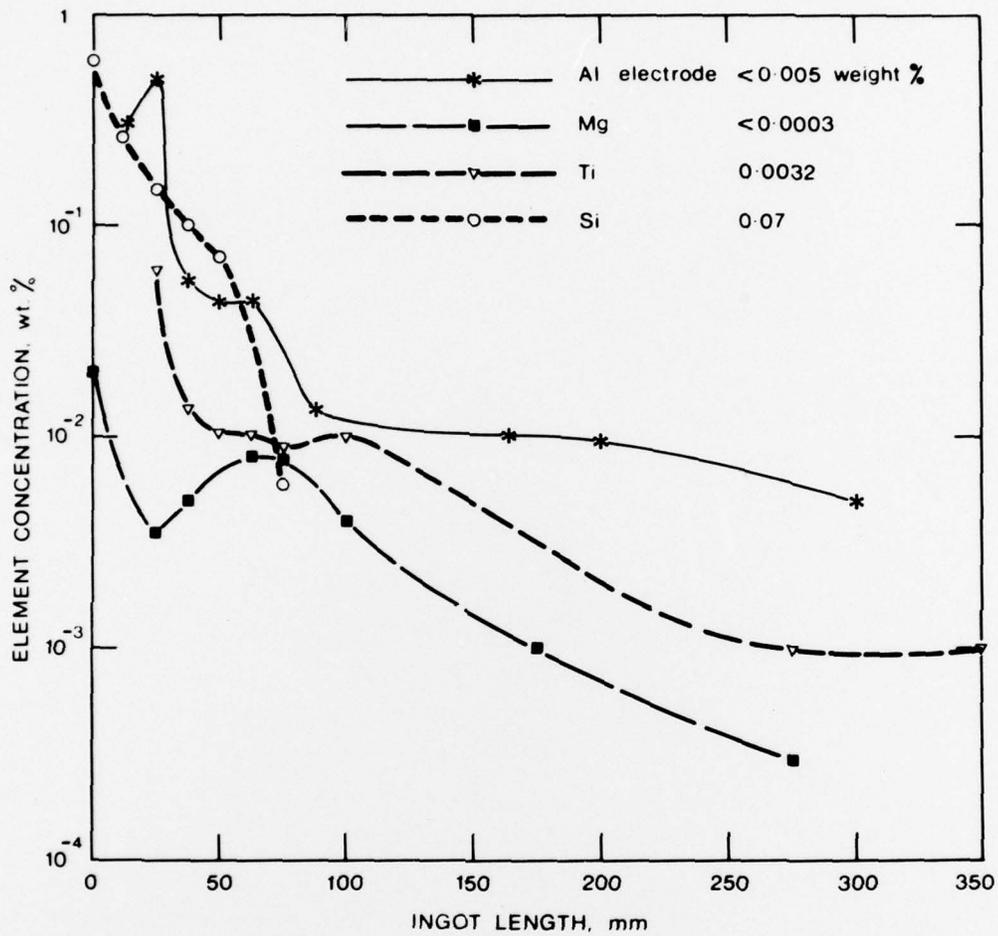


FIG. 6 - Extent of ingot contamination resulting from the use of the attachable exothermic starting compacts. Ingots were made with the electrode positive power mode.

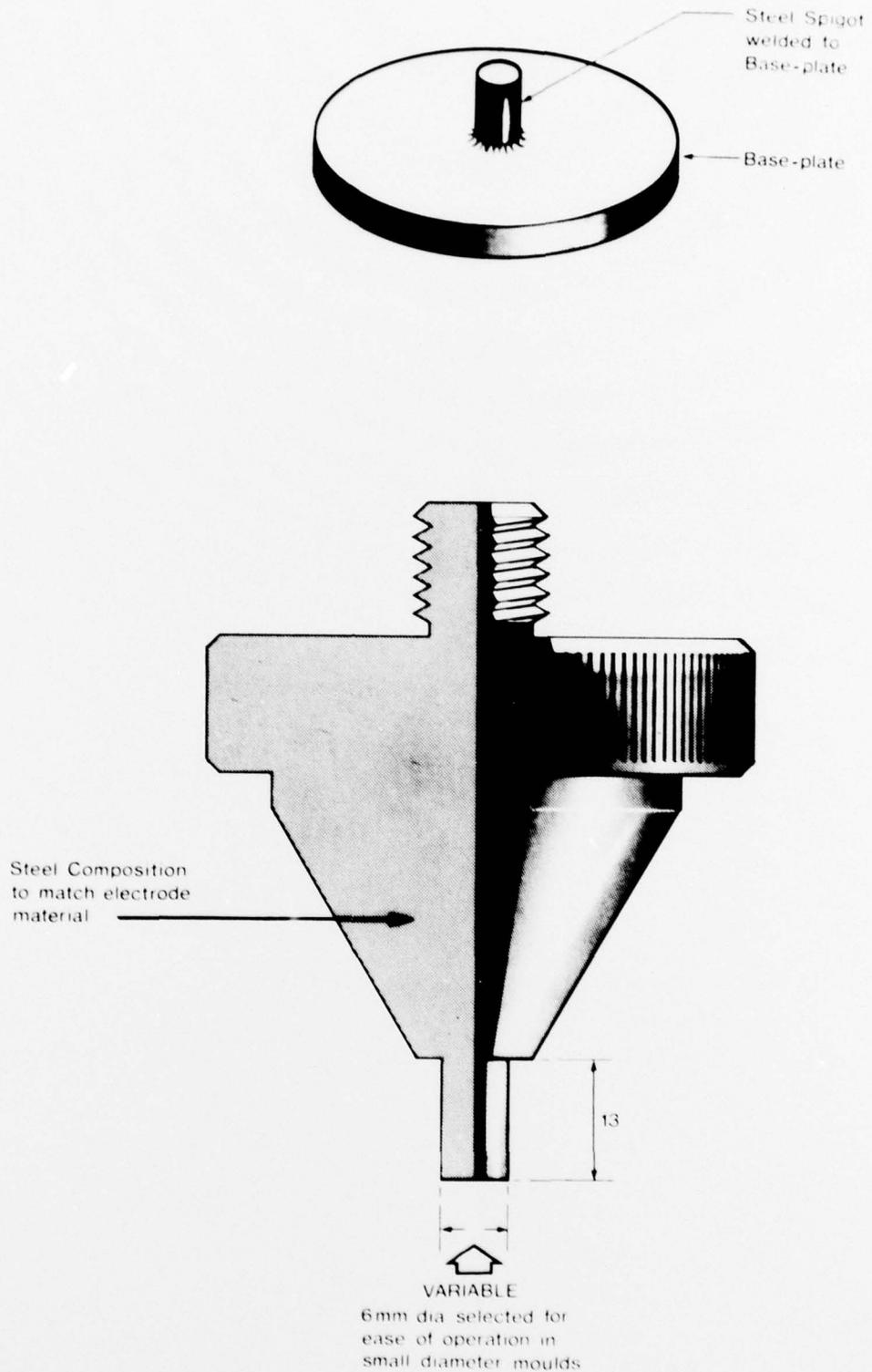


FIG. 7 - Resistance elements.

- (a) Steel spigot welded to the base-plate.
- (b) The steel core from the exothermic compact.

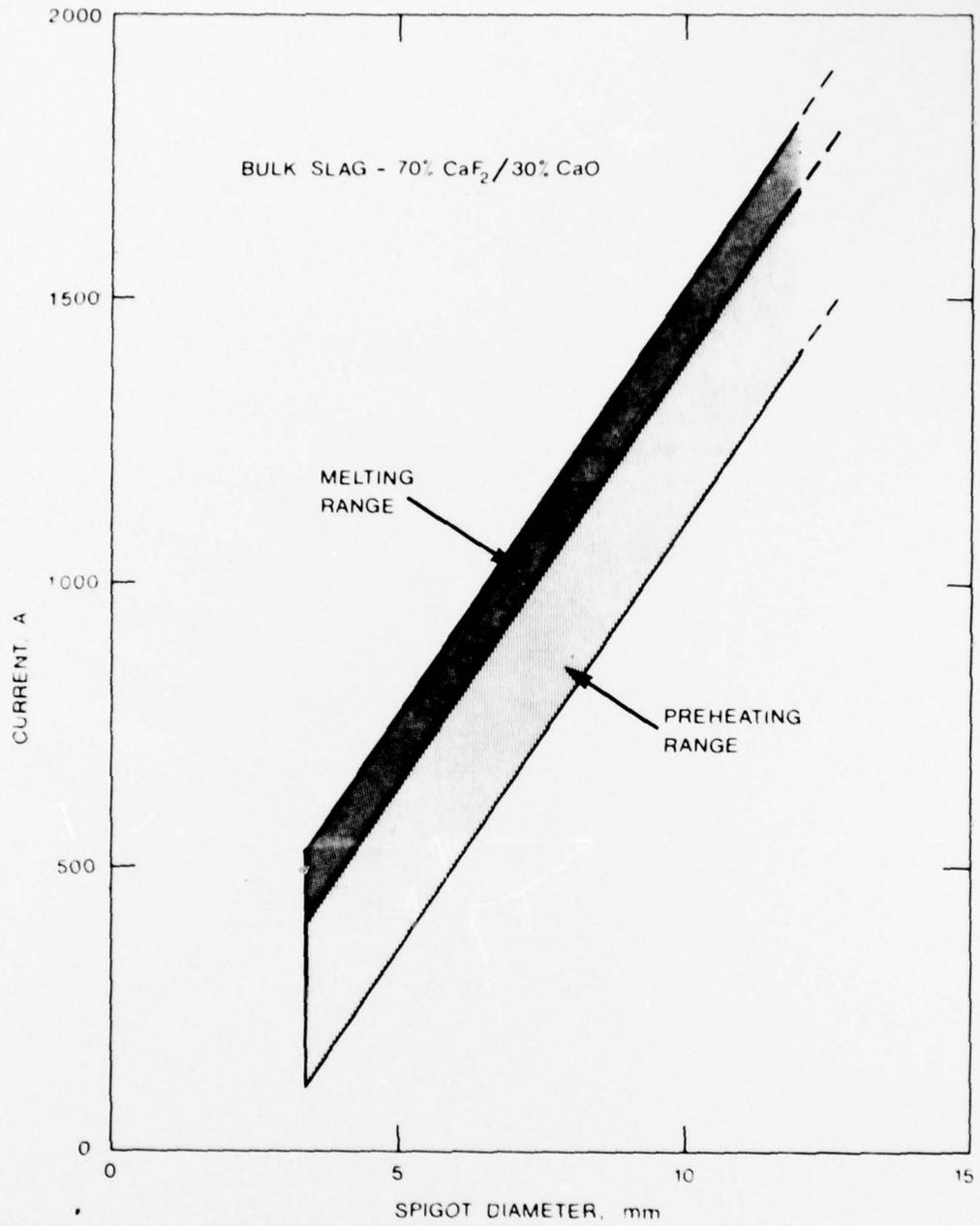


FIG. 8 - Preheating and melting currents for various diameter resistance elements.

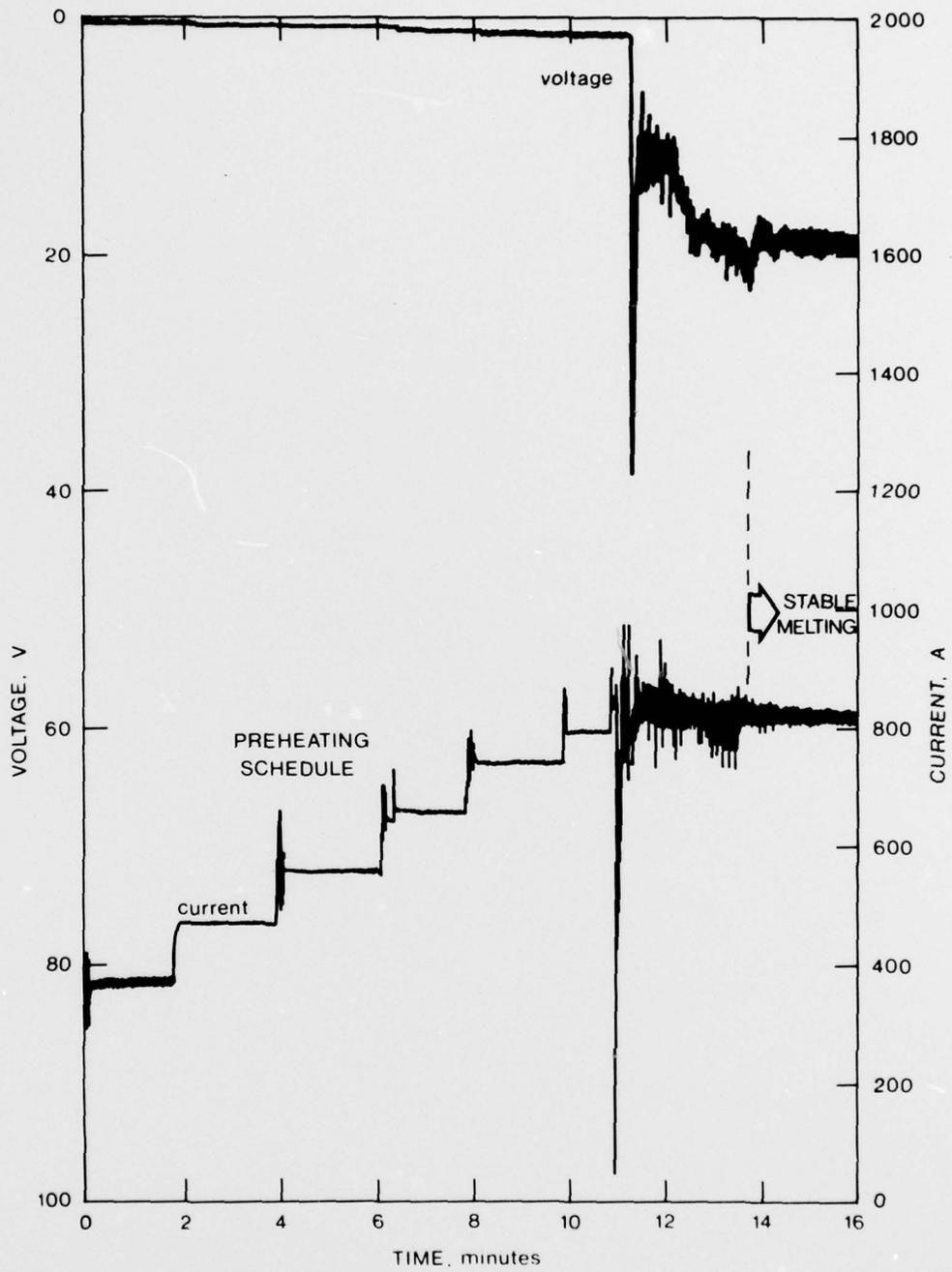


FIG. 9 - Current and voltage traces for a resistance start (6 mm dia. spigot).

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