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A Made in Brazil Metallic Sample Preparation Facility"

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We describe a facility, built locally, for the preparation of metallic compounds and alloys of common use in solid state physics. This facility includes a multipurpose furnace (FORARCO I) and accessories which are capable of melting, quenching, casting, and annealing.

Descrevemos um equipamento construido na UNICAMP para a preparação de compostos metálicos e ligas de uso frequente em física do estado sólido. Esse equipamento é composto de um forno de multiplas finalidades (FORARCO I) e de acessórios destinados à fundição, resfriamento rápido, produção de peças fundidas e recozimento.

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A large portion of contemporary solid state research concerns the properties of metallic compounds and alloys. The ability to prepare such materials is therefore of prime importance to innovative research. To facilitate studies in metallic magnetism and catalysis, a sample preparation laboratory was developed at UNICAMP[1]. The initial effort was the construction of FORARCO I, a multipurpose furnace installation built entirely in Campinas with special emphasis placed on the utilization of materials and components available in the Brazilian market. The FORARCO I installation consists of a versatile arc-melting chamber (see Figs. 1 and 2) as well as a high-vacuum chamber equipped to melt samples within resistively-heated refractory metal tubes (see Fig.3).

Arc-melting is a widely used laboratory technique for preparing metallic samples. Because of its simplicity, it is ideal for synthesizing compounds and alloys for exploratory experiments which often require the preparation of many samples. The sample components (usually pre-weighed portions of high-purity elements) are introduced into an inert gas atmosphere. There, confined within an indentation in a copper hearth [Fig. 1,e], they can be struck by an arc from a movable probe [Fig.1, a]. The arc passing through a sample generates Joule heat, and melting occurs if the arc current is sufficient. Any adjustable-output d.c. welding unit is an adequate power supply. The furnace described here supplied a maximum of 375 ampères at 35 volts (Ref.2), can melt samples with masses up to 20 grams at temperatures in excess of 3000°C.

Probe flexibility with vacuum integrity is provided by a bellows (beryllium-copper best; brass or stainless steel acceptable) [Fig.1, d]. The tip (thoriated tungsten welding rod) [Fig.1,c] embedded in the probe can thus direct an arc to a sample resting at any point on the hearth. It also serves as a finger, when the arc is off, to move sample or flip them. The portion of a sample contingent to the hearth is sometimes inadequately melted, so samples niay necessarily be flipped and re-melted several times to ensure homogeneity.

Since the hearth and probe are electrodes, the stainless steel base-plate [Fig.1, g] and aluminum top-plate [Fig.1, f], to which they are respectively attached, are insulated from one another by a thick-walled

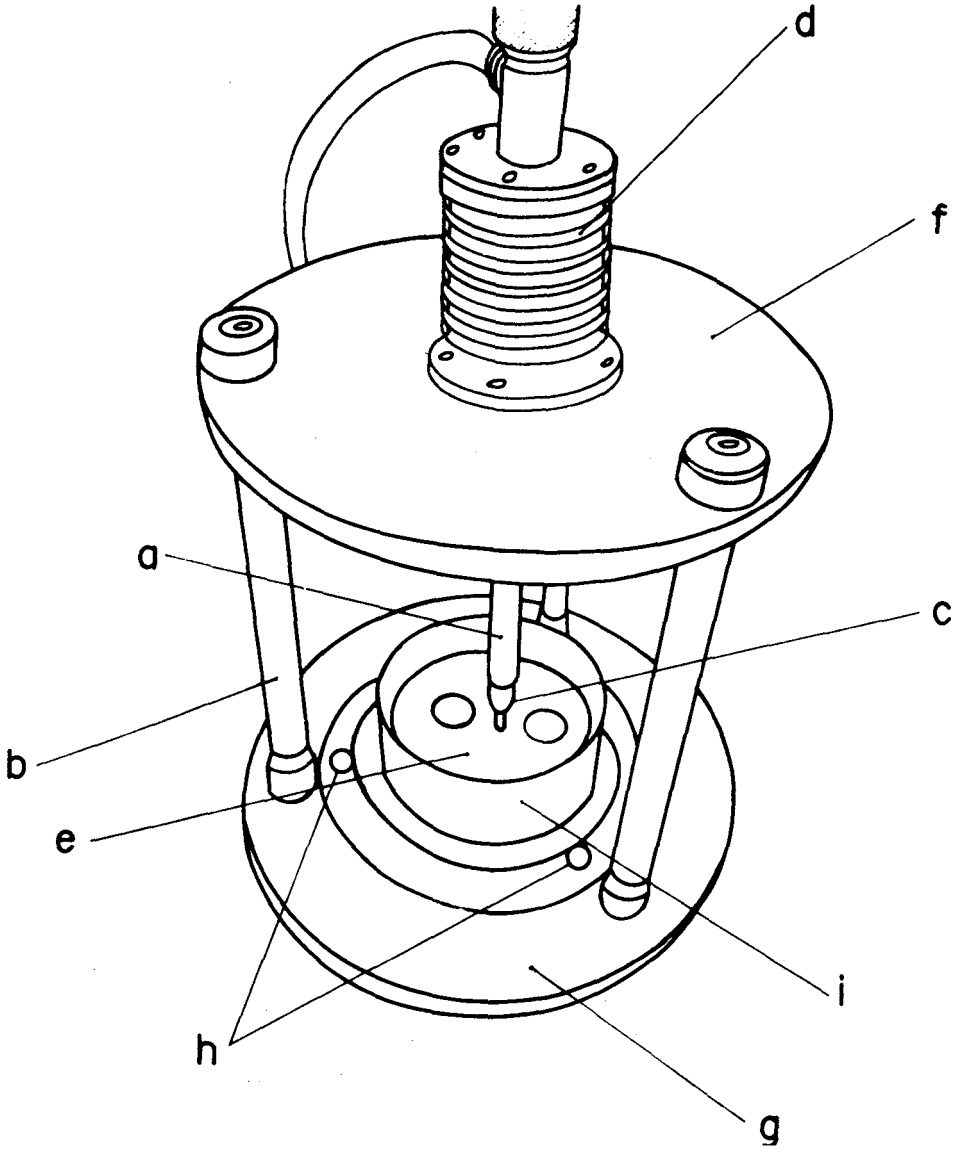

Fig. 1 - a. Movable probe; b. Non-conducting rod; c. Thoriated tungsten tip; d. Beryllium-copper bellows; e. Copper hearth; f. Aluminum top-plate; g. Stainless steel base-plate; h. Access ports; i. Copper radiation shield.

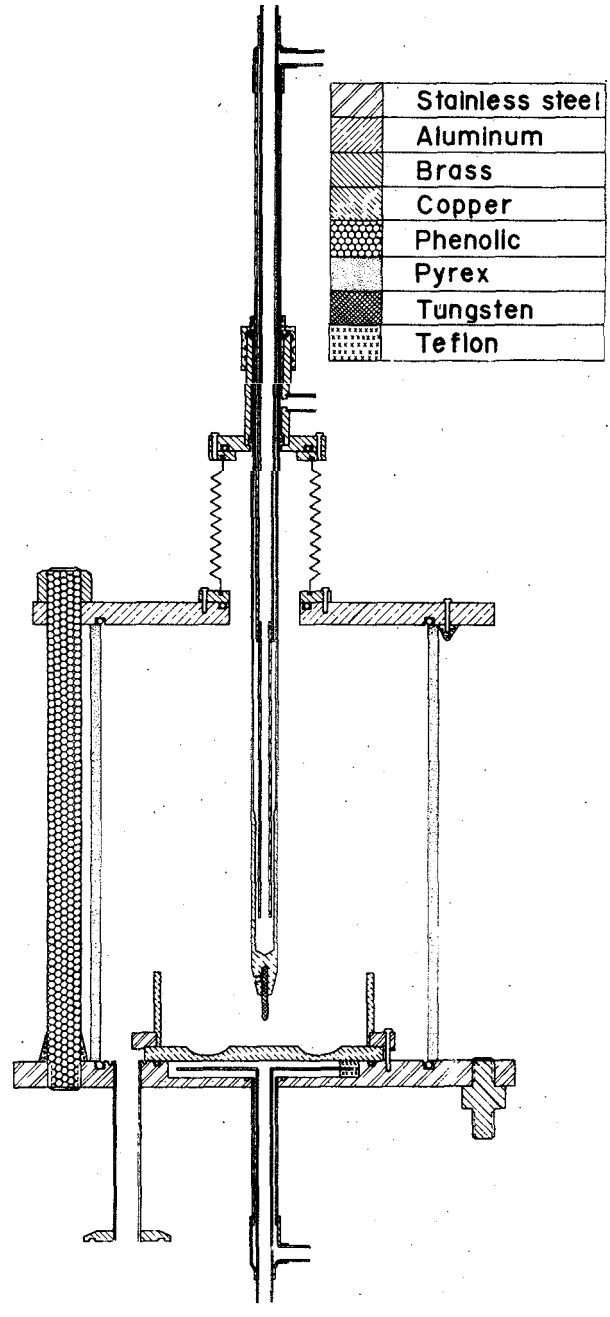

Fig. 2 - a. Cross section of the arc furnace.

pyrex cylinder (see Fig.2). This, of course, allows clear visibility into the furnace chamber. The ends of the cylinder, ground square, are bolted tighly against O-rings in the plate faces by non-conducting rods (e.g., phenolic) [Fig.1, b]. We note here two very important precautions: (1) the *probe* must be both *grounded* (since it is hand held) andne*gative* (so it collects any metal ions), and (2) *eyes* must be *protected* against intense radiation when viewing the arc. Surrounding the hearth is a radiation shield [Fig.1, i], a short section of copper tubing which serves to intercept the most intense radiation which might locally overheat the pyrex. The hearth and probe are water-cooled through the useof concentric tubing, copper fittings, and a baffle (see Fig.2). This minimizes sample contamination by metallic evaporation from parts of either electrode and eliminates the possibility of melting an O-ring or fusíng a sample to the hearth.

With a mechanical pump and a gas-handling system the furnace chamber can be evacuated and re-filled with an inert gas (argon or helium) through an access port [Fig.1, h). Pressure is monitored at another such port. In operation, the chamber is normally "pumped and flushed" several times prior to any melting. Further cleansing of active gases from the furnace chamber is achieved by melting first a piece of zirconium or titanium "getter". Discoloration of the getter is indication of a contaminated atmosphere; vacuum integrity and/or gas purity should be checked before proceeding to melt samples.

Ordinarily, arc-melted samples take the form of spheroids, flattened on one side by contact with the hearth; it is through this interface that they cool to room temperature. The basic arc furnace may be modified, however, to alter sample shape and rate of cooling. Two such modifications are described below.

With one modification a sample is quenched very rapidly from the molten state by splatting it against a clean copper surface. This can be achieved either by tripping a springloaded hammer which hits the molten sample or, more satisfactorily, by releasing a charge of pressurized inert gas which blows the melt against a wall (mechanical coupling or gas-line entry is through an access port). If the latter technique is employed,

som sort of over-pressure relief must be provided. "Splatt-quenching" is useful when attempting to retain a high temperature crystallographic phase which is lost during normal cooling.

With another modification, a sample can be cast into a rod[3] (suitable for a variety of measurements; resistivity or thermal conductivity, for example) by arc-melting it on a copper block over a hole (of desired geometry) which is opened suddenly to an auxiliary vacuum chamber (the vacuum line enters the furnace charnber through an access port). The molten sample solidifies as it is sucked into the hole. Heat-sunk to the water-cooled hearth, the copper block is constructed in halves, split at the hole, for easy retrieval of the molded rod. After "vacuum-casting" especially, annealing for strain relief may be necessary.

With either modification described above, a chamber still functions as an arc furnace. A different mode of operation, easily realized, permits more controlled melting of a sample. The procedure we will present is invaluable for reacting at high temperature components which have mismatched melting and boiling points[4]. Additionally, due to the existence of temperature gradients and the capability of careful temperature control, it is extremely useful for promoting single crystal growth. The sample components are placed in a refractory metal tube (usually tantalum) [Fig. 3,c] which short-circuits the probe [Fig.3,d] to the hearth [Fig. 3, e]. Should any component have a particularly high vapor pressure in the temperature range of sample formtion, the tube must be sealed (each end is flattened, then closed by an arc-melt weld; one before, the other after the sample components are introduced) before connection to the electrodes. For an open tube, electrical contacts are made simply by inserting a probe tip of appropriate diameter into one end of the tube and a tip embedded in the center of the hearth into the other; for a sealed tube, they are more elaborate. Melting is accomplished in an evacuated chamber (high-vacuum if desired - see below) by passing current through the tube, thus providing Joule heat surrounding the sample. The level of heating is adjusted by varying the output of the power supply. While a pyrometer may be employed to determine temperature, ordinarily one notes visually when a sample has melted. It wets the tube, hence there is a parallel current path across that region containing molten sample. Con-

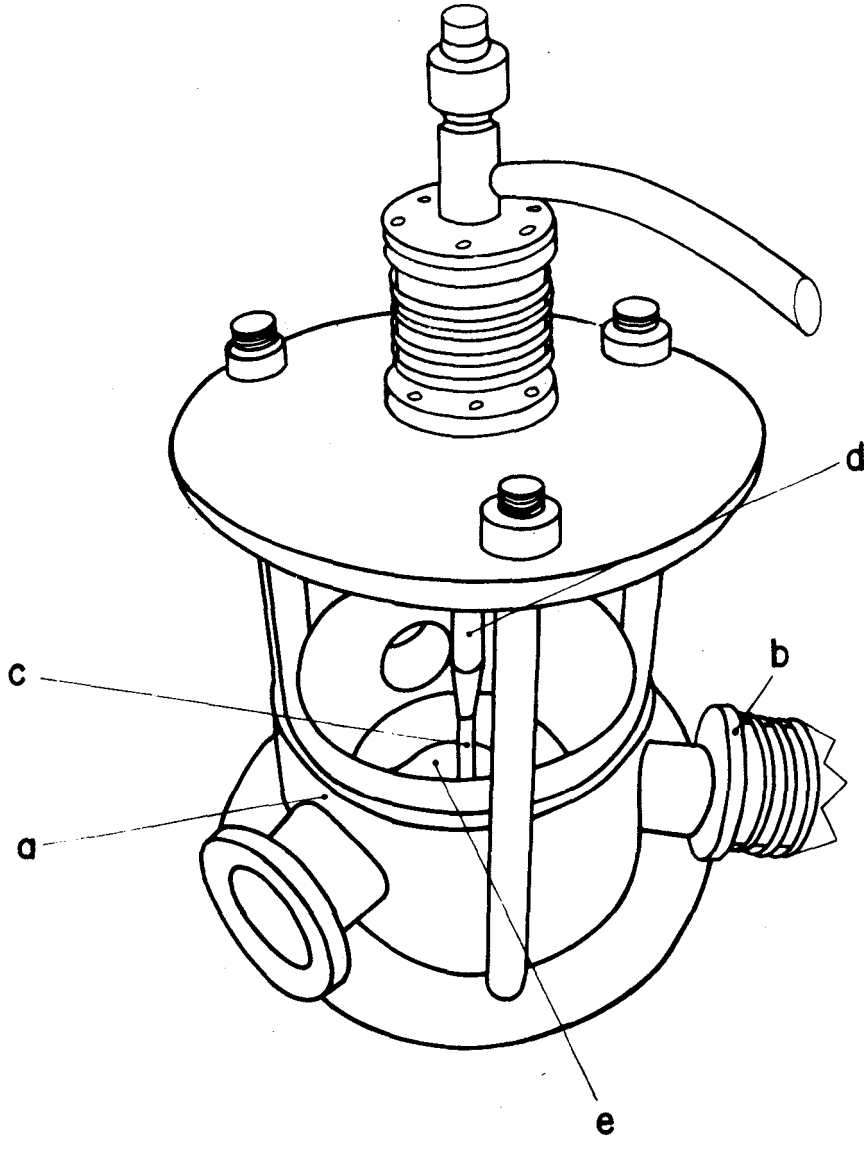

Fig. 3 - a. Brass collar; b. High-vacuum access port; c. Tantalum tube heating element; d. Water-cooled probe; *e*. Water-cooled hearth.

sequently, this area of tube is less heated and becomes darker than contiguous areas.

Since tube melting is done in vacuum and often over extended time (with crystal growth, especially), it is important that high vacuum (10^{-6} torr) be achievable within the chamber. To this end, a shortened pyrex cylinder is used with a collar [Fig.3,a], provided with access ports much larger than those in the base-plate. A diffusion pump is connected to one of these ports [fig.3,b]. The others can accomodate mechanical or electrical feed-throughs or, for that matter, splatt-quenching or vacuum-casting accessories.

Finally, we mention a supplementary installation and some additional techniques for metallic sample preparation presently in use at UNICAMP. We have built a modest glass bench equipped to evacuate, fill withinert gas, and seal off quartx tubes. Where tube-melting might be appropriate, but the sample forms below 1200°C and only moderate vapor pressures are involved, the components are sealed instead in a quartz tube with a partial atmosphere of inert gas. The sample is then reacted in a conventional oven. Where long-time annealing of an arc-melted sample is necessary to promote a particular crystallographic phase, to homogenize, or to strain relieve; again sealing in quartz in enert gas and oven-heating is employed. For annealing, a sample is wrapped in a refractory metal foil (customarily, tantalum) to avoid direct contact with the quartz, and a getter (titanium or zirconium foil or chips) is also included in the tube. A quartz tube, we note, can be withdrawn from an oven and plunged quickly, without breaking, into a water bath. Though not as rapid as splatt-quenching, plunging a sample sealed in quartz into water is another technique for retaining a high temperature crystallographic phase.

Together, FORARCO I and its accessories comprise an extremely useful and versatile metallic sample preparation laboratory. Furthermore, our facility represents a successful Brazilian effort to utilize local resources of material and manpower in the development of this country's technology. We will provide, in response to specific enquiry, detailed description of any apparatus discussed herein.

Much of the facility was inspired by one existing in the laboratory of

Professor Bernd Matthias at the University of California, San Diego. Two of the authors (J.G.H. and C.A.L.) gratefully acknowledge the technical assistance concerning sample preparation of Mr. Ben Ricks during the time they worked in the Matthias group. Another pair of authors (B.J.H. and J.G.H.) are responsible for a similar facility at Tufts University and they thank Mr. Walter Martin for his mechanical know-how there. At UNICAMP, we are all indebted to professor Carlos Rettori for his useful suggestions and general interest, and to Professors John Rogers and Marcos Zwanziger for their continuous encouragement. We also thank Mr. Gerson Ciampi for his technical assistance.

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