IS-3765

HIGH TEMPERATURE VACUUM-ANNEALING SYSTEM

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DISCLAIMER

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ABSTRACT

A system is described for use in homogenizing poly- and single crystal metal samples at temperatures up to about 1300°C. The furnace tube may be evacuated to a dynamic vacuum of 10^{-3} to 10^{-4} torr. A provision exists for pushing the sample from the hot zone into a zone the walls of which are at room temperature.

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In order to prepare chemically homogeneous specimens of solid alloys it is frequently necessary to heat cast ingots to a temperature just under the solidus and then hold them at this temperature for times as long as several hundred hours. In this connection, we have constructed an apparatus which has been used primarily to homogenize iron-base alloy single crystals at temperatures of the order of 1200° to 1300°C. The crystals are commonly 2.5 cm in diam. by about 7.5 cm long and are grown from the melt by a modified Bridgman technique.

A photograph of the apparatus is shown in Fig. 1. The retort is a 91 cm long by 3.8 cm i.d., 4.45 cm o.d. Al_2O_3 tube (McDanel, recrystallized, 99% Al_2O_3) with ground and polished ends. The furnace is 46 cm in length with a 35 cm heated zone and a 5 cm diam. bore. The Pt/40% Rh heating elements are non-inductively wound. The ends of the retort are maintained at a temperature near room temperature by close contact with hollow copper clam-shells through which tap water is continuously circulated. The vacuum system consists of a 500 liter/min. two-stage mechanical pump (not visible in Fig. 1) with a molecular sieve trap valve in-line. The pump and sieve trap are connected

by a flexible stainless steel hose. Excepting the connections at the immediate ends of the retort, all flanges in the system are copper gasketed. The manifold from the sieve trap valve to the left end of the retort is composed of the following stainless steel fittings: a nipple, a double-sided flange with a leak-to-air valve and a thermocouple gage, a cross (or tee), a bellows and a specially fabricated fitting. The left end of the latter has a 3.8 cm i.d. bore and is TIG welded to an 8 cm diam. flange. A 4.5 cm diam. concentric bore at the opposite end (2.5 cm deep with a square shoulder at the base) makes it possible to slip the fitting over the end of the retort. A viton gasket between the shoulder and the tube end provides for a vacuum seal. A similar fitting and gasket is used at the right hand end of the retort. Finally, this end is closed by a blank flange upon which is mounted two Conax feed-throughs. One of the latter accomodates a Pt/13% Rh thermocouple, the other a 3.2 mm diam. polished tantalum rod which is used on occasions to push crystals from the hot zone into the water cooled zone at the left end of the retort. Crystals are loaded/unloaded by removing the fitting/blank flange/feed-through assembly on the right.

With the central section of the retort at a temperature of about 1250°C, manifold pressures of near 3×10^{-3} Torr are achieved. This is satisfactory since our crystals are almost always encapsulated in evacuated quartz vials. If the viton gaskets are replaced by permanent metal-to-ceramic seals (Torr Seal has been

very effective for this purpose) and the flange with feed-throughs is replaced by a plain blank flange, then manifold pressures of at least 2×10^{-4} torr are reached under conditions similar to those just given.

We are grateful to Mr. Gaylord Stowe for assistance in the construction and operation of this apparatus, and to Mr. Alex Litwinchuk for designing and fabricating the support assembly.

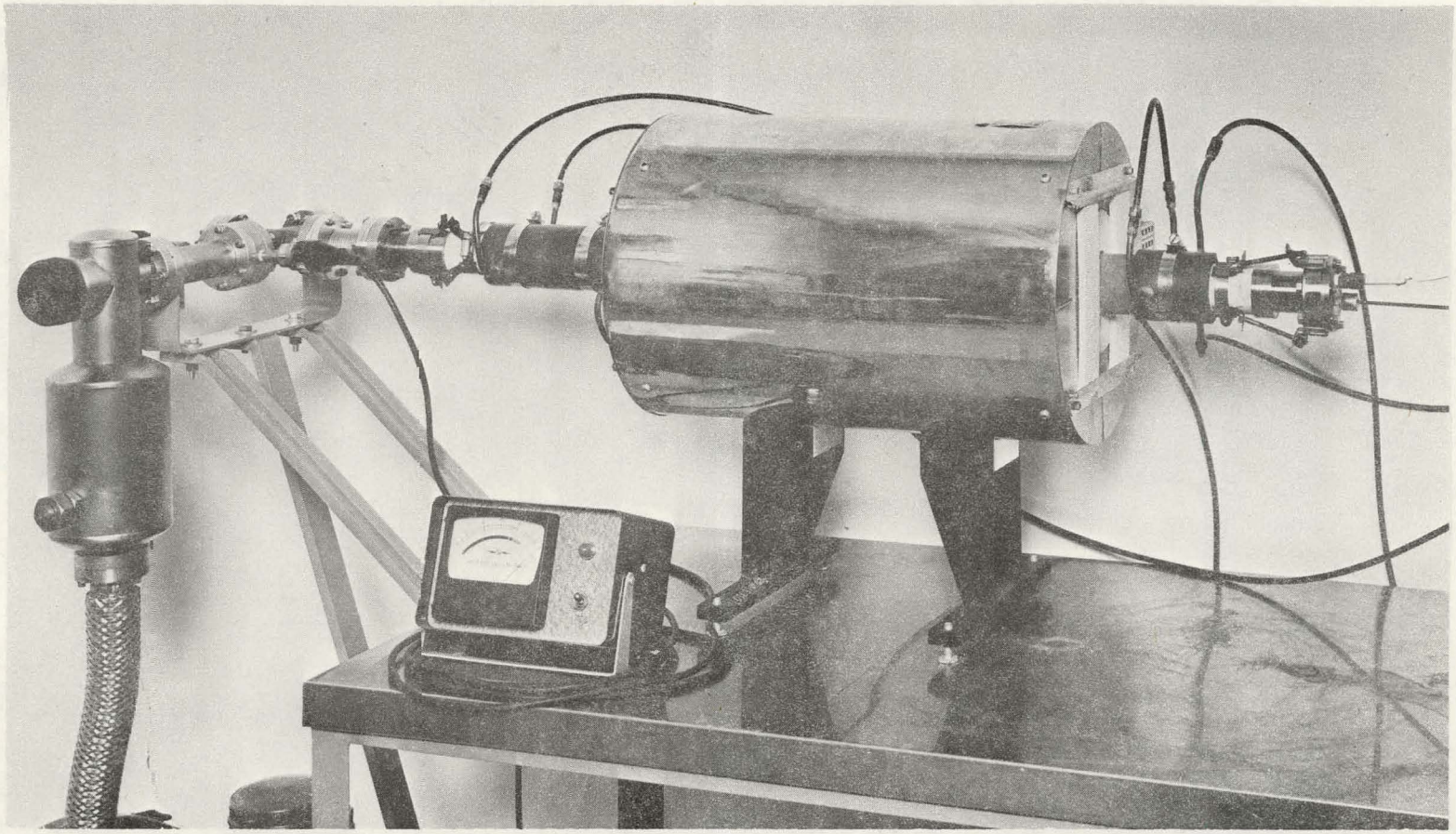

Fig. 1. High temperature furnace and vacuum system