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High Temperature Vacuum Furnace for the Preparation of Graphite Targets for ^{14}C Dating by Tandem Accelerator Mass Spectrometry

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ABSTRACT

A simple and reliable furnace design capable of producing temperatures of up to 2800°C is presented. The furnace has been specifically designed for the rapid and reliable production of graphite targets for ^{14}C dating purposes but may be used in a variety of applications requiring high temperatures under vacuum conditions.

KEYWORDS

TAMS;

C-14;

VACUUM FURNACES;

GRAPHITE;

TARGETS;

RADIOCARBON DATING

1. Introduction

The radiocarbon dating method traditionally uses radioactive decay counting techniques to determine the amount of ^{14}C present in samples. In the last few years however increasing use has been made of particle accelerators as ultra sensitive mass spectrometers to determine ^{14}C. (1,3) In this method a sputter ion source is used to liberate C^- ions from a solid carbon target derived from the original sample material to be dated. These targets are usually made from crystalline graphite because this material produces intense stable beams of C^- ions when placed in an ion source. The ions are counted for ^{14}C, ^{13}C and ^{12}C thus enabling ^{14}C dating of the original sample. The new method offers several advantages over conventional decay counting techniques. In particular extremely small samples containing 1 mg or less of elemental carbon may be dated(4). However before the method, known as Tandem Accelerator Mass Spectrometry (TAMS), can be fully utilised suitable techniques for converting typical sample materials into solid graphite targets are required. We have developed a quick and reliable method for producing graphite targets(5,6). The technique uses temperatures in the vicinity of 2200°C to induce a phenomenon known as catalytic graphitisation in which amorphous carbon is transformed into three dimensionally ordered G effect graphite(7). To achieve these temperatures a small high temperature, vacuum furnace was designed and built in this laboratory. The design is relatively simple and the purpose of this paper is to provide a description of the construction and operation of the furnace.

2. Experimental

The furnace consists of a hollow graphite element 90 mms long with an internal diameter of 7 mms. It is fashioned in the shape of a "dogbone" and mounted in graphite sleeves held in two stainless steel, water cooled terminal posts (see figure 1). The element is readily turned on a lathe from a 1/2" piece of Union Carbide high vacuum, ECV grade, graphite rod. Care is required to ensure that the wall thickness of the "hot zone" is about 0.7 mm. The thin section has a relatively high resistance which leads to lower power consumption at high temperatures and rapid dissipation of surplus heat from the furnace. The furnace element and terminal posts are contained by a stainless steel cap lined with a tantalum radiation shield. The cap is fitted with a quartz observation window which is used with an optical pyrometer to determine the operating temperature of the furnace. The cap is vacuum sealed to a stainless steel base plate with a viton "O" ring and the apparatus is evacuated through a filter and vacuum port as shown in figure 1.

To use the furnace the cap is removed and the sample to be graphitised is placed into the "hot zone" portion of the furnace element. The sample is generally contained in a tantalum or boron nitride boat some 20 mm long and 5 mm in diameter, sealed with a plug having a 0.5 mm hole to allow gas to escape. Some tests have also been made in small graphite boats although these are not desirable for ^{14}C dating because of the risk of contamination of the sample by graphite from the boat. The sample boat is wedged into the "hot zone" with small pieces of 2 mm diameter graphite rod. This prevents the possible rapid evolution of gas from the sample from moving the boat out of the "hot zone". During the heating process the rate of heating is adjusted to keep the pressure in the furnace tube below 0.1 Torr. Experience has shown that, depending on the sample type, too fast a heating rate can result in the boat rupturing with subsequent loss of sample.

The furnace element is connected to the secondary of a 6 kw transformer rated at 12 VAC and 500 Amps. The primary of the transformer can be driven by either a 25 Amp, 230 VAC varian or a solid state power supply. Because of the large physical size and the high cost of large variacs we have found it convenient to use a variable power supply built around a conventional 100 watt 230 VAC light dimmer and a 16 Amp Triac. The light dimmer is used to drive the gate of the triac which regulates the amount of current delivered to the primary of the transformer. Under typical graphitising conditions at 2150°C the furnace element draws about 200 Amps at 7 volts from the secondary, well under the 6 kw rating of the transformer. Transformer primary currents are typically of the order of 7 to 10 amps and are easily controlled by the light dimmer triac system.

Typically a complete cycle producing a graphite sample suitable for ^{14}C analysis by TAMS takes about 10 minutes .... a 5 minute heating stage, from room temperature up to 2150°C, about one minute at 2150°C to complete the catalytic graphitisation and four to five minutes cooling to room temperature.

3. Conclusions

The furnace design presented here provides a quick and convenient apparatus for preparing graphite target material suitable for ^{14}C dating by TAMS. Although the furnace has been specifically designed for this purpose it can be used for any application where temperatures of up to 2800°C under vacuum conditions are required. When a larger sample size is needed the graphite sleeves are removed from the terminal posts and a bigger graphite furnace element used (see figure 1). In our experience larger furnace elements do require more heat to be dissipated by the furnace cap but this can be achieved simply with a pair of fans providing a forced draft or a small water cooling coil attached to the furnace cap.

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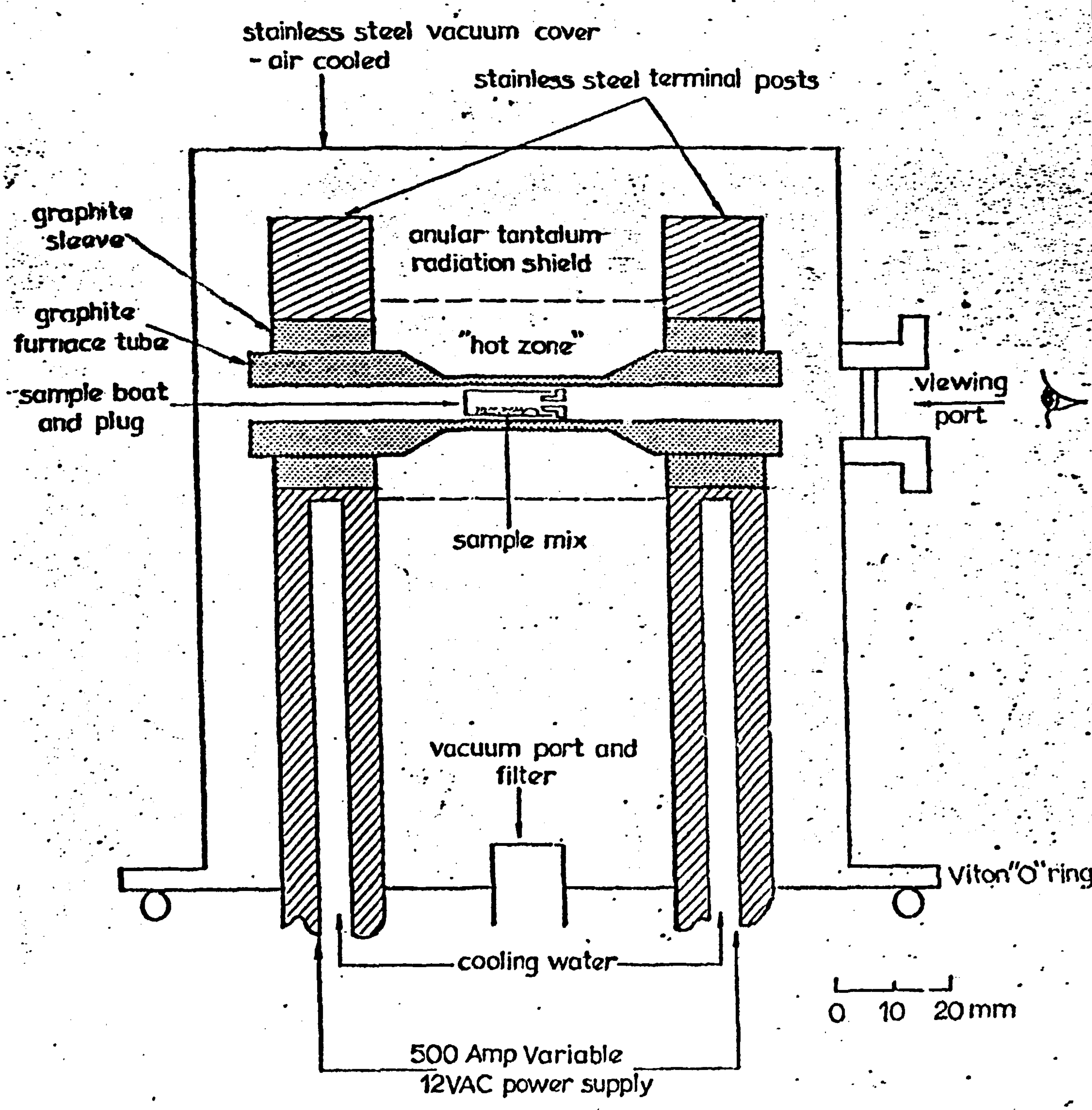
stainless steel vacuum cover
- air cooled
stainless steel terminal posts
graphite sleeve
anular tantalum radiation shield
graphite furnace tube
"hot zone"
sample boat and plug
viewing port
sample mix
vacuum port and filter
Viton"O"ring
cooling water
0 10 20mm
500 Amp Variable
12VAC power supply