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THE USE AND EXTENDED LIFETIMES OF MICROFURNACES FOR THERMOGRAVIMETRY. PART II. REPAIR OF BROKEN FURNACE STEMS

C.M. EARNEST and DWIGHT KINZER

Department of Chemistry, Berry College, Mt. Berry Station, Rome, Georgia 30149 (U.S.A.)

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ABSTRACT

A procedure is described for the repair of mullite stems which support the wound furnace-mandrel used in microfurnaces which are employed internally in the furnace tube of the TG system. Three different degrees and variations of furnace damage are noted and their repair described. Materials used for the furnace-stem repair are described so that the reader may also employ the repair procedure.

INTRODUCTION

In a previous paper [1], the use and construction of microfurnaces which are employed in a particular vendor's thermogravimetric apparatuses were described. The advantages and disadvantages of the popular low-mass furnace were discussed. Finally, some recommendations for cleaning the furnaces and, hence, extending their lifetimes were made. In this paper, we address a common problem encountered by users of one of the older model TG systems employing this internal microfurnace.

REMOVAL OF THE PYREX FURNACE TUBE FROM THE TG FURNACE AND FURNACE-SUPPORT ASSEMBLY

The small internal microfurnaces employed in the modern thermogravimetric systems sold by the Perkin–Elmer Corporation are held in an upright position in the furnace tube by a mullite stem which is about four inches long. The mullite stem is surrounded by short support posts at its base and fits flush into a stainless steel assembly which is machined to fit into a ball mount in the furnace-support assembly. All of these features are clear in Fig. 1, which is a line drawing of the furnace, outer Pyrex furnace-tube enclosure, and furnace-support assembly used in the Perkin–Elmer TGA 7 thermogravimetric system.

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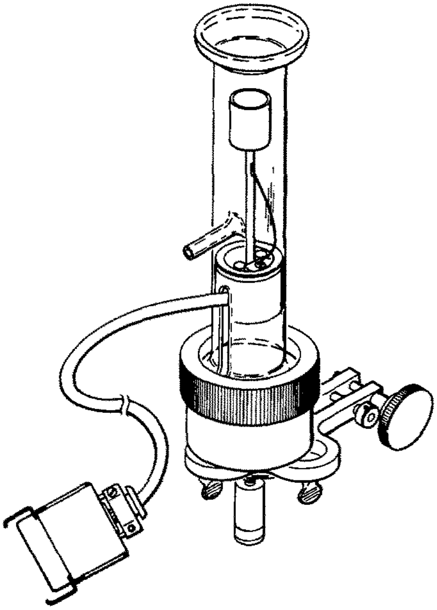

Fig. 1. Drawing of TG microfurnace, outer pyrex furnace tube, and furnace-support assembly used in TGA 7 system.

There are occasions when the surrounding Pyrex furnace tube must be removed: these are primarily for the replacement of either the furnace or the small stainless-steel-sheathed thermocouple which is located in the furnace assembly. In the latest model, the TGA 7 thermogravimetric analyzer, this is easily accomplished by simply loosening a furnace-locking nut and adjusting the furnace height-positioning knob while the furnace is in the "down" position. The Pyrex furnace tube is then released from a split O-ring at its base and the furnace tube separates from the furnace and furnace-support assembly. This is a greatly improved procedure for removing the furnace tube relative to that which is required for the older model TGS-2 thermogravimetric system. In the TGA 7 there is little, if any, opportunity for breaking the supporting mullite furnace stem while removing the Pyrex furnace-tube enclosure from the furnace assembly.

With the older TGS-2 system, the procedure for lifting the outer Pyrex furnace tube from the furnace-support assembly is not so straightforward. In the TGS-2 system, the Pyrex furnace tube is held tightly at its base by an unsplit O-ring. The operator's manual [2] for this system recommends that the furnace tube be clamped at the top by a pinch-clamp (see Fig. 2), then, after loosening a locking nut at the base of the furnace tube, the furnace-support assembly is lowered with the furnace assembly in place. The furnace tube is separated by pulling it past the tight O-ring which is part of the furnace-support assembly. The successful execution of this procedure results

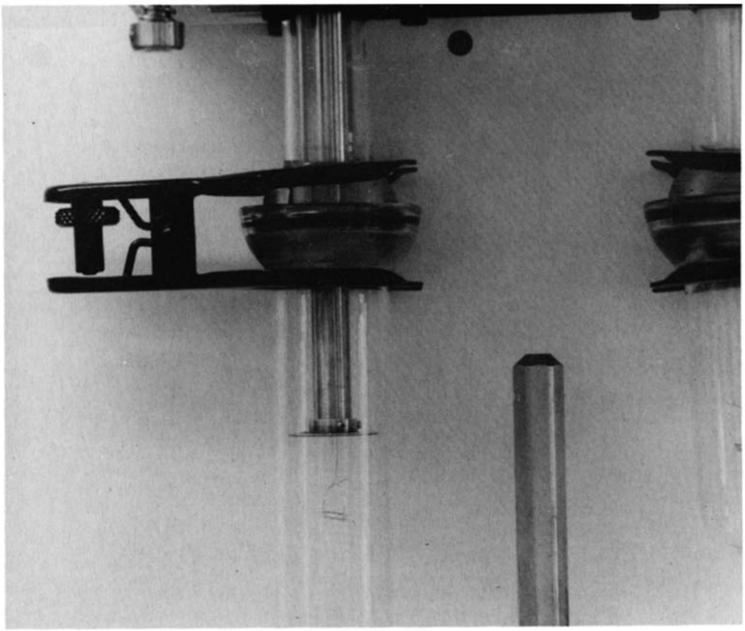

Fig. 2. TG pyrex furnace tube clamped to ball joint prior to separation of tube from furnace-support assembly.

in the separation of the furnace tube from the furnace and furnace-support assembly as shown in Fig. 3.

Some operators prefer to simply remove the furnace tube by giving it an upward pull while the furnace is in the "down" position. This can be achieved if the O-ring at the base of the furnace tube is sufficiently loose (this condition is often achieved by removing and stretching the O-ring) to allow the forced separation of the Pyrex tube from the furnace-support assembly. Unfortunately, on initial installation, the O-ring is meant to be vacuum tight and, hence, is strongly held by the furnace-support assembly. In many cases, when sufficient force is applied, the furnace tube is finally pulled away in something less than a completely vertical manner. This can cause the furnace tube to intersect the stand-up microfurnace, usually resulting in damage to the furnace stem. This is the most frequent cause of furnace-stem fracture or dislocation but some operators have managed to achieve the same result when using the previous method recommended by the user's manual [2]. Once the supporting mullite stem is damaged and the furnace does not stand upright in a near vertical manner, the sample pan and stirrup cannot be positioned into the small furnace by a vertical wire. Hence, the microfurnace, even though it will still operate properly as a heating device, is rendered useless for the purpose of performing thermogravimetry.

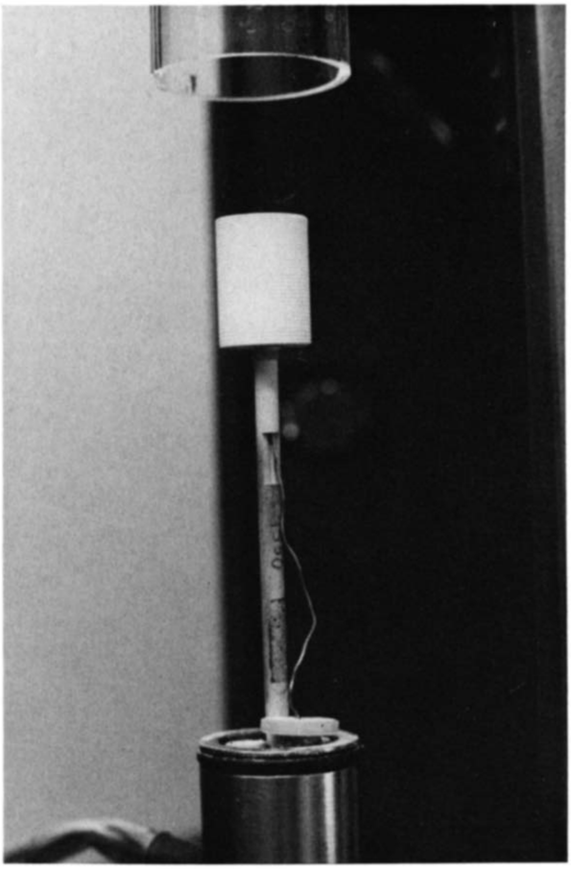

Fig. 3. TG microfurnace and TG furnace tube (above furnace) after execution of the separation procedure.

REPAIR OF BROKEN MICROFURNACES

This study was made possible by obtaining damaged furnaces from several operators at laboratories using the same thermogravimetric system which we employ, the Perkin–Elmer TGS-2. Because this is one of the most popular TG instruments ever sold, it was not difficult to find users with broken furnaces. Our laboratory contributed two broken furnaces to the study, a result of inexperienced students attempting to replace the standard furnace tube, which has one arm (the purge-gas outlet), with a two-armed furnace tube used for purge-gas switching during TG studies.

Because the replacement cost for the furnace is about $700.00, there have been many different approaches used to mend the damaged mullite stem and to restore the furnace to an upright position for use in the thermogravimetric analyzer. Because the mullite stem itself does not get nearly as hot as the furnace mandrel during the actual furnace operation, various types of glues, etc., have been used by some frustrated operators to restore the furnace to use. This is often a temporary restoration as volatile decomposition products and heat can deteriorate the glue bonding of the mullite stem. In the following paragraphs, we suggest a procedure which we consider to be the best approach to restoring microfurnaces with damaged mullite stems to a condition suitable for performing thermogravimetry.

Classification of damaged furnaces

We have categorized the broken microfurnaces into three different groups depending on the type of stem damage incurred. The first type of damaged furnace might well be the most commonly encountered and represents the least problem to the operator. This is shown in Fig. 4 and has been named "basal dislocation of the furnace stem". In this case, contact with the furnace tube during removal has separated the base of the mullite stem from the stainless steel assembly such that the furnace is no longer perfectly

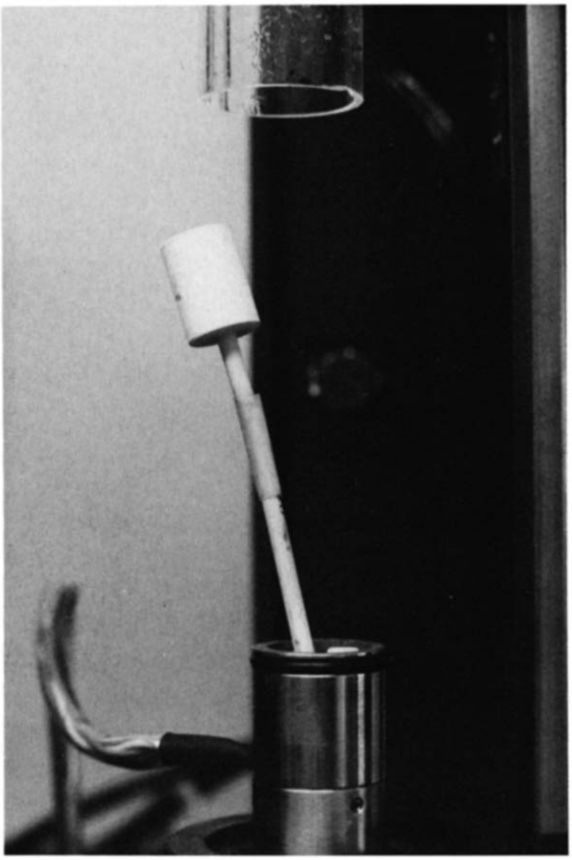

Fig. 4. Broken TG microfurnace suffering from "basal dislocation of furnace stem".

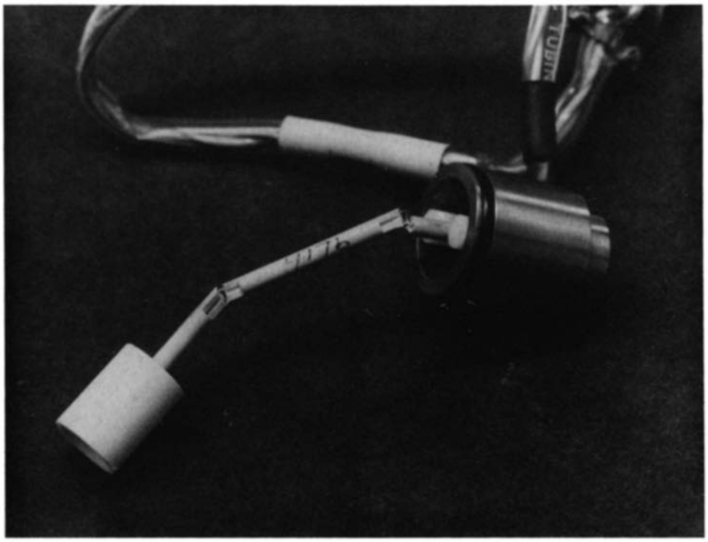

Fig. 5. Broken TG furnace suffering from "severe cumulative damage" (SCD).

vertical (90° to the bench surface). The vertical wire, stirrup and sample pan cannot enter the furnace nor can the Pyrex furnace tube be placed around it without constant contact with the furnace.

The second category of furnace damage is described as "stem fracture". In this case, the mullite stem is broken somewhere between the points where the mullite stem joins the furnace mandrel and where the stem enters the stainless steel base-assembly. Obviously, this type of damage may be either simple (one break) or compound (more than one break) for a particular furnace stem. Our repair procedure, given below, has been effective in repairing both simple and compound fractures. The furnace shown in Fig. 4 not only suffers from basal dislocation but also has a simple stem fracture which for photographic purposes is held together, at near mid-height of the furnace, by several rounds of Scotch Tape.

Obviously, the third group of broken furnaces is the most severely damaged and possess both stem fractures and basal dislocation of the mullite stem. Figure 5 includes such a microfurnace which we have categorized as suffering from "severe cumulative damage" (SCD). As bad as this damage may appear, the procedures described below will generally repair these furnaces as well.

REPAIR: METHODOLOGY AND PROCEDURE

All of our repairs employed a mullite tube with on outer diameter of 0.25 in (6.27 mm) and with a wall thickness of 0.88 mm, obtained from Coors

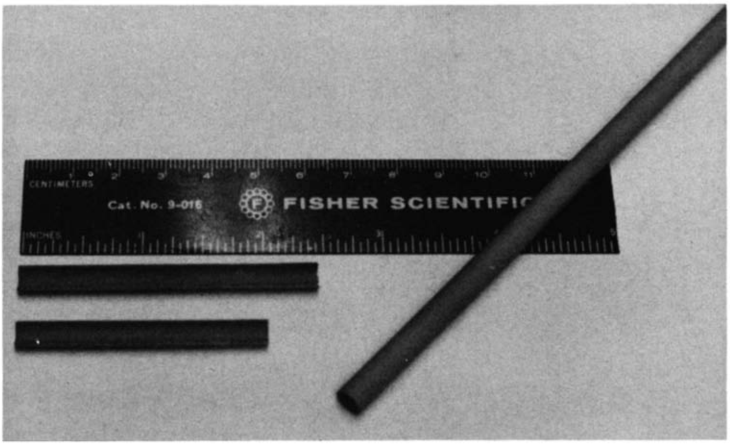

Fig. 6. Mullite tubing and two mullite sleeves (50.8 and 61.0 mm) cut from the tubing and used in the repair procedure.

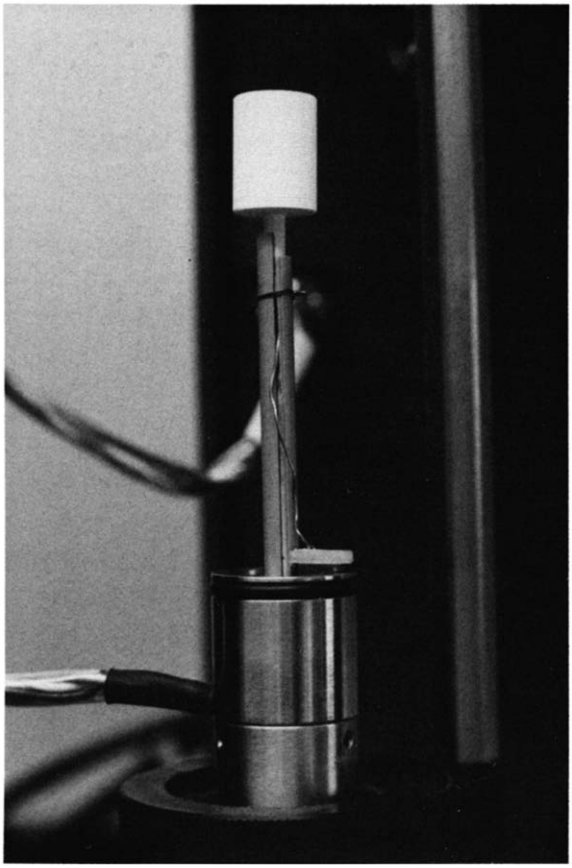

Fig. 7. Repaired TG microfurnace.

(Golden, Colorado) in 12 inch lengths. The mullite tubing was cut into the proper lengths which were then split into two halves in order to facilitate furnace repair. The cutting and splitting of the mullite tubing was achieved by a high speed hand grinder (Sears) employing a cut-off wheel of diameter 1 in. The cutting stock of mullite was cooled by a jet of water as it was cut. Figure 6 shows a portion of the original mullite tubing together with halves of two different lengths of split mullite tubing: the plastic ruler is shown for scale.

We have found that we can repair all furnaces from the above categories by choosing one of two sets of different lengths of split mullite tubing: 2.0 in (50.8 mm) and 2.4 in (61.0 mm). Both lengths are shown in Fig. 6.

For repair of furnaces suffering only basal dislocation, the 2.0 inch (50.8 mm) split tubing is all that is required for complete repair. In this case, the mullite 'sleeves' are fitted between the basal support posts and the furnace stem. The mullite sleeves are then wired together at the top with 26 gauge wire (Pt is preferred) such that it does not interfere with the placement of

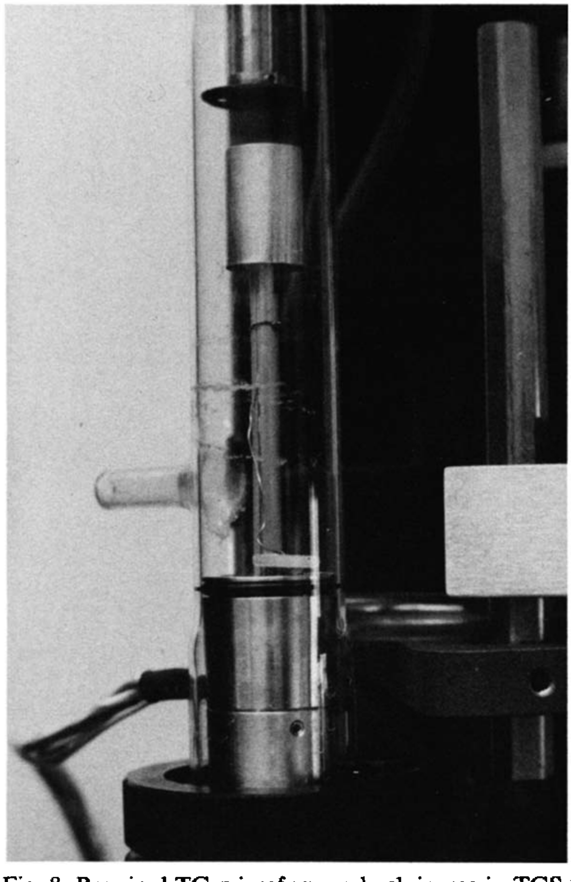

Fig. 8. Repaired TG microfurnace back in use in TGS-2 system.

the measuring thermocouple. The two halves of the split mullite tubing are arranged so that the thermocouple entry slit in the original furnace stem is available to the thermocouple.

Figure 7 shows a furnace which has been repaired with cut mullite tubing secured in place at the top with 26 gauge (0.40 mm) copper wire. The two sleeves are positioned so that the thermocouple enters the usual orifice for its placement in the furnace. The furnace shown here suffered from simple stem fracture as well as basal dislocation prior to repair with the mullite sleeves.

The choice of length of the mullite sleeves (either 50.8 mm or 61.0 mm) depends on the position of stem fracture. Furnaces having stem fractures low on the stem may require only 50.8 mm sleeves and thus the repair procedure is the same as that for those furnaces having stems with basal dislocation only. On the other hand, furnaces having stem fractures in the vicinity of the thermocouple orifice, or higher, require the use of the 61.0 mm sleeves to achieve the repair.

Figure 8 shows a repaired TG furnace in use inside the Pyrex TG furnace tube. This represents the ultimate goal of the repair effort. To date we have repaired a total of 6 broken furnaces which is all we have had available to us. We believe the repair procedure is applicable to all broken furnaces of this type. The only requirement is that the lead wires to the furnace mandrel must not be broken.

CONCLUSION

The proper length and diameter of mullite tubing, when split into two symmetrical sleeves, may be used to repair broken microfurnaces. Thus, perfectly good furnaces which have been rendered useless by broken support stems can be salvaged.

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