27 cyp.
5/8/75
cyp. to NTL

2

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RE-ESTABLISHING FILTERING CAPABILITIES OF MACHINED POROUS
BERYLLIUM VIA CHEMICAL REDUCTION AND CLEANING

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May 1, 1975

MASTER

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RE-ESTABLISHING FILTERING CAPABILITIES OF MACHINED POROUS BERYLLIUM

VIA CHEMICAL REDUCTION AND CLEANING

By William L. Randall

ABSTRACT

Porous Beryllium is furnished in sheets of varying sizes and thickness, it is therefore necessary that it be machined into specified sizes. A chemical reduction and cleaning procedure has been devised to remove the disrupted surface, open the sealed pores of the material and clean entrapped contaminants from the internal structure. Dimensional stability can be closely controlled and material size is of no consequence.

INTRODUCTION

When porous Beryllium is machined the pores of the material become sealed, fluids used in the machining operation are entrapped and the surface is disrupted to quite a depth. It is necessary that these pores be opened, that entrapped fluids and other foreign matter be purged from the external structure, that the disrupted surface be removed and dimensional stability be maintained.

The requirements of the finished parts are fairly rigid and it was determined that the best methods for meeting them was by utilizing chemical techniques. The reasons for this determination are three-fold:

1. Material removal can be closely controlled.
2. Rapidity of the process
3. Development of a new solution for the chemical polishing and etching of Beryllium that will achieve the first two requirements.⁽¹⁾

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Experience has proved that material treated by this method has met all requirements and has been chemically clean throughout the porous structure.

SAFETY PRECAUTIONS

The procedure must be performed in a chemistry hood, and safety glasses or a face shield, industrial rubber gloves, and a laboratory coat or apron must be worn. The solution becomes toxic when beryllium is dissolved in it and hydrogen gas is involved. The gloves must be checked for cuts or small holes since it may sometimes be necessary to put one's hands in the solution. The solution is very stable and can be used for a great number of parts, but it should be bottled between uses. When it becomes necessary to dispose of the solution, it must be bottled, identified as to content and turned over to Hazards Control for disposal. Cleaning agents such as Acetone and Methanol are highly flammable and laboratory safety regulations must prevail when they are used.

CHEMICAL REDUCTION AND CLEANING EQUIPMENT AND PROCEDURES

EQUIPMENT

The chemical reduction equipment consists of the following items:

1. A chemistry hood, preferably one with a sink and flowing water so that spilled drops of solution are well diluted before they enter the drainage system.
2. Pyrex beaker or jar for solution - 2000 ml or larger
3. Hot plate for heating solution
4. Rinse water
5. Hot plate for boiling distilled water
6. 2 beakers for boiling distilled water - depending on part size
7. 3 beakers for solvents - depending on part size

8. Tongs, tweezers, or other devices for holding part
9. A stop watch
10. A micrometer for measuring material removal
11. White gloves
12. A clean area for cleaned parts
13. Vacuum oven for bake-out of finished parts

SOLUTION

The solution should be made up in sufficient quantity to fill the solution jar and to replace any solution lost by evaporation, spilling or adherence to parts. The formula for the solution is as follows:

By volume:

50 parts - $\text{CH}_3\text{CHOH}\cdot\text{COOH}$ (89 to 90%)

40 parts - H_3PO_4 (85 to 86%)

10 parts - H_2SO_4 (96 to 97%)

All acids should be C.P. grade

TEMPERATURE

This solution has a very wide temperature range and can be used above 100° C without becoming unmanageable. No breakdown of the solution has been observed.

REMOVAL RATE

The removal rate is steady and is directly related to temperature. At temperatures between 65 and 70° C, the removal rate is .0005 in. (0.0127 mm) per minute. At temperatures between 80 and 85° C the removal rate is .001 in. (0.0254 mm) per minute. This is a steady and calculable rate, but it can be affected

by the build-up of beryllium in the solution.

Temperature should be constantly monitored and time controlled by stop watch. Material should be removed from solution, rinsed, dried and measured at regular intervals. This function also allows the contact points of the holding device to be shifted so that etching is uniform and no pits, ridges or streaking are allowed to form.

PROCEDURE

Parts should be cleaned thoroughly in Acetone to remove all surface contamination caused by the machining process. With the solution at temperature the material is submerged in the solution for a measured period of time. The material is then removed, rinsed, dried and measured. These steps are repeated until the pores of the material are exposed.

The entrapped contaminants will start seeping from the internal structure. These contaminants can be forced from the material in the following manner:

1. Remove material from the etch solution
2. Rinse well in cold running water
3. Rinse well in hot running water
4. Take a Micrometer reading
5. Place material in boiling distilled water and allow to boil until surface is free of bubbles.
6. Remove quickly and plunge into Acetone. The Acetone will be sucked into the air free structure.
7. Remove material from the Acetone and place into the boiling water. The Acetone will boil out of the pores carrying contamination with it.

8. Steps 5 through 7 should be carried out 3 times
9. Place material in etch solution. At one minute intervals, or whenever it is deemed necessary to check dimensions, steps 1 through 8 should be repeated.
10. When the final dimension is reached, steps 1 through 8 should be repeated and in addition the material should be cycled from boiling water into Methanol 2 times and cycled from boiling water into Freon TF 2 times.
11. The final Freon TF cycle should be followed by a good boiling in clean distilled water.
12. If contamination such as surface weeping or other foreign matter is detected after step 11 is completed, the Freon TF cycle should be repeated until all contamination is purged from the material.
13. When material is clean internally, wash the material well in C.P. grade methanol and bake in a vacuum oven for one hour at 100° C.
14. Upon removal of material from vacuum oven, check well for any signs of contamination. If contamination is detected, repeat cleaning procedures and re-bake.

DISCUSSION

This procedure is written as a general guide. The techniques employed are fairly well known and call only for conscientiousness on the part of the person performing the task. The cleaning operation is critical to the final integrity of the material and short cuts are to be avoided.

NOTE: It is important that in the dimensioning of porous beryllium parts allowance is made for this procedure.

REFERENCE:

1. Invention Case No. IL-5685 - William L. Randall

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