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NASA TECH BRIEF

Marshall Space Flight Center

Fabrication of Porous Plugs for Control of Liquid Helium

The problem:

Porous-plug phase separators prevent the unnecessary loss of liquid helium stored in Dewars. The plugs, which are inserted in the vent lines, must have high thermal conductivity and small pore size, approximately 1 to 2 μm in diameter. Currently used plugs have high thermal conductivity, but the pore size ranges between 10 and 15 μm. On the other hand, plugs with pore diameters in the 1-μm to 2-μm range are available with ceramic materials which have very poor thermal conductivity.

The solution:

Copper plugs can be made with pore sizes ranging between 1 and 2 μm using a newly-proposed fabrication method.

How it's done:

The new method takes advantage of certain properties of copper and combines two well-known procedures: oxygen annealing and hydrogen annealing. Copper has a very low free energy of surface oxidation: If copper is heated at an oxygen pressure of 10^{-3} torr or less, a surface oxide is not formed, but instead oxygen diffuses into the bulk to form a solid solution. However, impurities in the copper with a higher free energy of oxidation will be oxidized. This process of oxygen annealing is commonly used to produce copper of high electrical purity.

Under certain circumstances, these oxidized impurities can be made to precipitate at the grain boundaries. Oxides of copper can also be made to precipitate at the grain boundaries. If such copper is subsequently heated in hydrogen, the hydrogen diffuses into the copper, reacts with the precipitated oxides, and produces separations at the grain boundaries. These separations form the necessary pores.

The proposed fabrication procedure is as follows:

a. Copper with the desired grain size is prepared by the selected addition of impurities and appropriate heat treatment.
b. The copper is machined to the desired size and shape, and then it is electrochemically polished to remove surface damage caused by the machining.
c. This part is then oxygen annealed. Precipitation at the grain boundaries is controlled partially through the choice of impurities and the regulation of oxygen pressure during anneal, but it is controlled primarily through control of the anneal temperature.
d. If the plug is a disk, an additional refinement at this stage is possible. The plug as a whole will be larger after firing in hydrogen than before firing because of the grain boundary separation. A ring of OFHC (oxygen-free, high-conductivity) copper can be machined and placed around the disk in the furnace before heating in hydrogen. The inner diameter of the OFHC ring will be slightly less than the outer diameter of the disk after the hydrogen anneal. Then upon firing, the plug will expand into the OFHC ring and thus will be permanently mounted in a retainer ring with an expansion coefficient perfectly matching that of the plug at all temperatures. The plug can then be installed by standard techniques (soldering, indium O-ring seal, or the like) without changing the effective cross section of the plug.
e. The final step is the hydrogen anneal.

In summary, by the consecutive annealing of copper in two different gases, porous plugs can be fabricated with the desired pore size. The ratio of channel area to metal area is controlled through the control of grain size, and total channel area is controlled by controlling the oxide precipitation at the grain boundaries.

(continued overleaf)

Note:

Requests for further information may be directed to:

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Patent status:

Inquiries concerning rights for the commercial use of this invention should be addressed to:

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