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A VARIABLE-VOLUME SILVER LINER FOR HIGH
PRESSURE HYDROTHERMAL CRYSTAL GROWTH

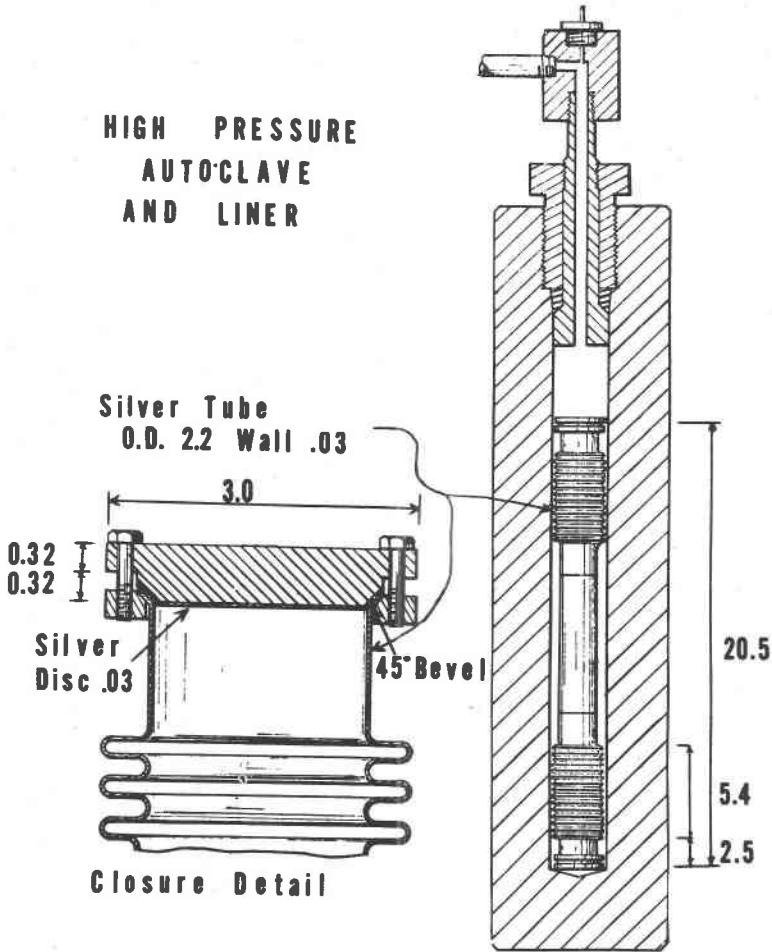
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ABSTRACT

The design and use of a variable-volume silver liner for hydrothermal crystal growth is described.

We wish to report details of the design, construction and use of a variable-volume silver liner for high pressure autoclaves. It has proved especially suitable for hydrothermal growth of crystals such as zinc oxide and potassium zinc phosphate. They are best grown from a basic medium like aqueous potassium hydroxide which is extremely corrosive to steel at elevated temperature. Although the use of noble metal liners or plating in autoclaves is an established practice (Ballman, 1963) it has been attended with serious problems including fabrication, sealing and pressure equalization.

The liner illustrated in Figure 1 was designed for use with the high pressure autoclave shown there and described in the experimental section. Silver bellows segments are formed from dead-soft cup stock by hydraulic pressure applied internally. As the tube expands outward into a die, it is folded longitudinally, forming bellows. Sealing of both tube ends is accomplished with a silver disc flared to conform to the flared end of the tube. A circular



**All Dimensions In
Centimeters No Scale**

FIG. 1. Variable-volume silver liner.

mild steel collar bears against the outer flared surface of the tube, while a tapered mild steel plug bears against the outer surface of the flared silver disc. The collar and plug are drawn tightly together with twelve steel machine screws (size 0-80), thus sealing the two silver surfaces.

The intrinsic advantage of this liner lies in the fact that the percent fill in the liner may be adjusted without opening the liner. Thus, in practice the liner is charged with nutrient, a seed, and crystal growing medium such as potassium hydroxide solution. After sealing

the liner is placed in the autoclave, and the annular space between the liner and autoclave is charged with sodium hydroxide solution and about one-half gram of silica. In this way a layer of acmite, a sodium ferric iron silicate, is formed on the internal surfaces of the autoclave making them impervious to attack from the aqueous solution up to 425°C. There is no attack at this temperature. The autoclave is sealed, and the system brought to proper temperature. During heat-up the liner bellows can expand or contract if a pressure difference develops between the internal fluid and the annular fluid. With proper selection of the percent fill of the liner and the annular space, the change in linear dimension of the liner is negligible. The final pressure can be adjusted as desired by addition of fluid to the annular space with a fluid pump or by withdrawal of fluid.

The liner has several additional advantages. Heliarc welding is not required to seal the liner, and the flared silver disc and steel collar, plug and machine screws are easily replaced. The liner can be removed easily from the autoclave, facilitating repairs and charging and clean-up procedures. The design allows scale-up to any size autoclave, making commercial application feasible. The exposure of dangerously corrosive potassium hydroxide solution is confined to silver surfaces only.

Silver liners as described have been used successfully hundreds of times up to 400°C for the hydrothermal growth of crystals of potassium metal phosphates and related substances. A wide variety of metals including platinum and gold can be formed into bellows and thus lend themselves readily to the basic design. Physical and chemical requirements of the system under investigation together with economic considerations would dictate the choice of material.

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NICKELIAN MACKINAWITE FROM VLAKFONTEIN, TRANSVAAL: A DISCUSSION

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I would like to present some published and new observations on naturally-occurring mackinawite, which complement those of Vaughan (1969), who has described an unusually nickel-rich specimen of this mineral from the Transvaal.