Chapter 5

Conclusion

The objective of this thesis work was the preparation of periodically precipitated copper oxalate. Pre-forms in the system of agar gel and glass powder were used to start with. The systems can be produced in one characteristic ratio. The suitable ratio is 30 ml agar gel (0.3 g agar + 30 ml water) per 100 g glass powder. At this ratio, agar gel can fill all voids of the glass powder preform while the glass powder is statistically packed. Making the glass powder hydrophobic is a major prerequisite for success of the consecutive precipitation step. With hydrophobic glass powders, the problem of heterogeneous nucleation encountered in gel systems containing ceramic particulate matter is overcome. Thus multilayered precipitates can be prepared like in pure agar gels containing $Cu(NO_3)_2$ as outer electrolyte solution and $(NH_4)_2C_2O_4$ as inner electrolyte solution. In this thesis work, three hydrophobic agents were studied, which are: silicone oil, dimethyl-dichlolo-silane, and paraffin wax. Silicone oil was the best suited coating agent, presumably replacing the hydroxyl group of the

glass surface by $(-SiCH_3CH_3-O-)_n$ chains. But a serious problem occured. The coating could not be removed by heat treatment, but rather decomposed to white SiO_2 at the outside and black Si at the inside, because the oil has a considerable oxygen deficiency. Dimethyl-dichloro-silane and paraffin wax were chosen for coating the glass surface because both of them can make the glass surface hydrophobic. The silane makes a chemical coating while the paraffin wax makes a physical coating increasing the surface active (water repelling) area. The electrolyte concentration suitable for periodic precipitated was in a narrow range, 0.2 - 0.3 M $Cu(NO_3)_2$ and 0.1 M $(NH_4)_2C_2O_2$. As volume of outer electrolyte, 1.00 ml is more suitable than 2.00 ml. The preforms can be made in the shapes rods, hollow rods, or torus. Glass powder with smaller particle sizes (63 to 125μm) makes the rings occur earlier than powder with bigger sizes (125 to 180 μm). The spacing of precipitates can be controlled by the pH value of the inner electrolyte. Higher pH values lead to larger spacing. It is demonstrated by screening tests that the precipitates can be partially decayed into metallic copper, and that the precipitates can be incorporated in the glass matrix by viscous sintering.