Synthesis, characterization and high temperature corrosion of glass-ceramics in the B_2O_3-Al_2O_3-SiO_2 system

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Introduction

A limitation in the use of materials containing boron oxide, is the B_2O_3 volatilization at high temperature. In dry atmosphere and at atmospheric pressure, the B_2O_3 volatilization starts around 1000°C (1). Nevertheless, this phenomenon is enhanced in presence of moisture and becomes very important at temperatures lower than 1000°C. Boron oxide reacts with gaseous water to produce metaboric acid HBO_2 and orthoboric acid H_3BO_3 with high vapor pressure. Then the glass network (B-O-B bonds) is broken, causing the borate phase volatilization and the rapid degradation of the material.

The aim of this study is to reduce boron oxide volatilization under high temperature and corrosive atmosphere. The stabilization of boron oxide can be achieved by addition of an element X, which can lead to a definite compound aX_mO_n-bB_2O_3, expected to be stable in wet atmosphere at high temperature. For this purpose, aluminum is incorporated in order to form $2Al_2O_3.B_2O_3$ and $9Al_2O_3.2B_2O_3$ (figure 1). The purpose of this work is in a first part, to synthesize and to characterize glass-ceramics in the B_2O_3-Al_2O_3(-SiO_2) system and, in a second part, to study their behavior under wet atmosphere at high temperature.

Results and discussion

Synthesis and characterization of glass-ceramics

Glass-ceramics were prepared by mixing oxide powders B_2O_3, Al_2O_3 and SiO_2 with different contents. Due to its moisture sensitivity, B_2O_3 reacts with H_2O to form HBO_2 and H_3BO_3 at room temperature. To avoid the loss of boron by volatilization, the mixtures were first heated slowly up to 250°C and maintained 3h at this temperature for dehydration. The samples were then heated up to 700°C, 800°C or 900°C for 3h in air in an opened Pt crucible and quenched in water. The $2Al_2O_3.B_2O_3$ compound was obtained only after a heat treatment at 900°C according to the reaction [1] :

$$2Al_2O_3 + B_2O_3 = Al_4B_2O_9 \qquad [1]$$

Such an aluminum borate was identified by XRD analysis. By SEM observations, it was found to form as whiskers. For compositions including SiO_2, a borosilicate glass was also formed after a heat treatment at 900°C, as shown by infrared analysis.

Behavior under wet atmosphere at high temperature

The synthesized samples were tested at 900°C or 1200°C with respectively a 30kPa or 50kPa water vapor pressure, obtained by bubbling N_2 or N_2/O_2 in hot water. It must be noted that for a temperature beyond 1035°C, a peritectic reaction with formation of the other definite compound $9Al_2O_3.2B_2O_3$ is observed (reaction [2] and figure 1).

$$2Al_2O_3.B_2O_{3(s)} = 9Al_2O_3.2\,B_2O_{3\,(s)}+\text{liquid} \qquad [2]$$

The ratio of the measured weight on the initial weight of B_2O_3 is drawn versus time and for various Al_2O_3 contents respectively in figures 2 and 3. The results show that the $2Al_2O_3.B_2O_3$ and $9Al_2O_3.2B_2O_3$ compounds are able to catch a part of boron oxide through their stability in water vapor at high temperature. The addition of silica also inhibits the volatilization of boron oxide.

Conclusion

The entrapment of boron oxide by alumina, as well as the presence of SiO_2, reduce the volatilization of boron oxide at 900°C and 1200°C under corrosive atmosphere.

Acknowledgements

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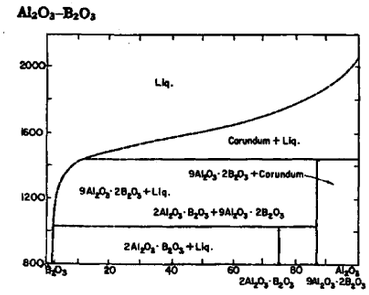

Figure 1 : Phase diagram Al_2O_3-B_2O_3 (wt %)

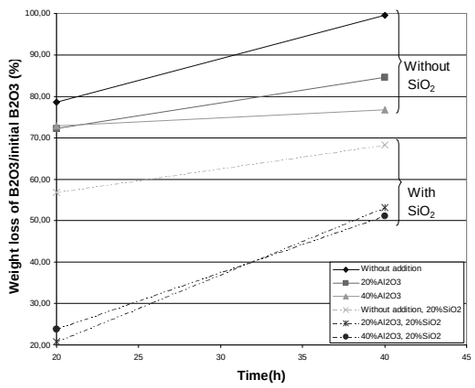

Figure 2 : Time variation of B_2O_3 weight loss at 900°C under a 30kPa water vapor pressure with or without addition of Al_2O_3 and SiO_2

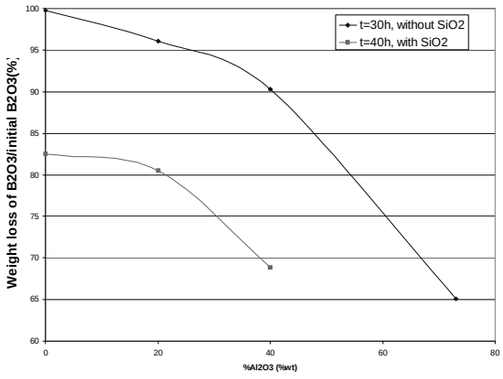

Figure 3 : Weight loss of B_2O_3 at 1200°C under a 50kPa water vapor pressure versus the Al_2O_3 content in the initial mixture

(1) J.R Soulen, P.Sthapitanonda, J.L Margrave, « Vaporization of inorganic substances : B_2O_3, TeO_2 and Mg_3N_2 », vol **59**, p.132, 1954.