Synthesis of sintered glass-ceramics in air and argon obtained from industrial waste revealed by hot stage microscopy (HSM)

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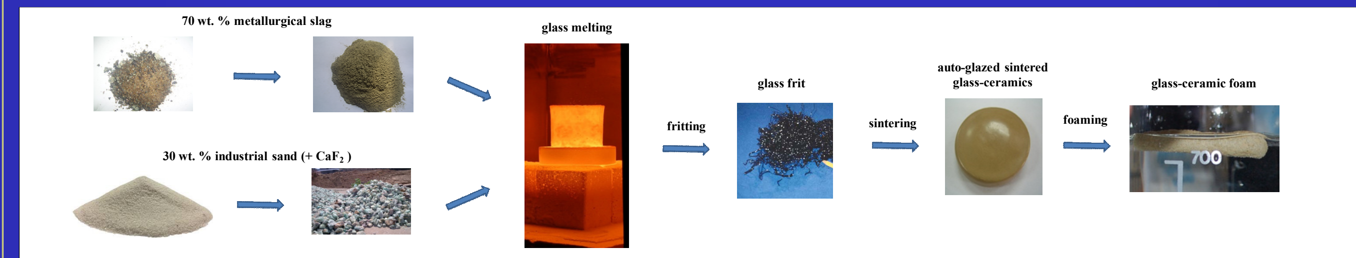

Introduction

Many industrial streams in the metallurgy provide large amounts of waste raw materials, e.g., in the form of slag, containing the necessary composition allowing its entire recycling with the introduction of just minor modifications. In this way new glass-ceramic materials can be obtained. The used technology includes melting and subsequent sinter-crystallization treatment usually carried out in atmospheric environment. The used raw material contains iron oxides, which is a valuable property. These oxides are known to act as foamers at certain known conditions, i.e. they can initiate the formation of new foam materials. The existing dependence of the linear heating rate on the sintering behavior during the formation of new glass-ceramic materials can be considered as the motivation of present investigation.

Recent implementation of innovative synthesis in air and argon or in a mixed air/argon environment used by the authors, has led to increase of the degree of sintering and to a stronger kinetic effect when carrying out a stage of the synthesis of sintered glass-ceramics in argon instead of air.

Results & Discussion

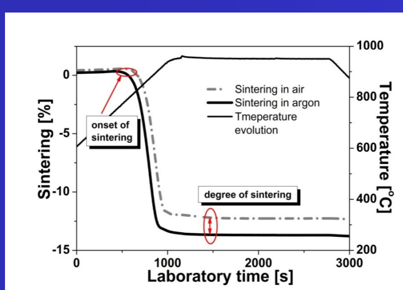

Figure 1a: Sintering curves measured in air and argon

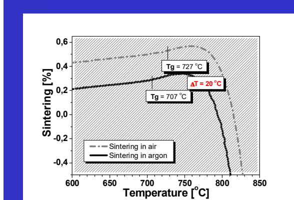

Figure 1b: Sintering curves; zoom-in of the circled area of Figure 1a. The glass transition temperature (*Tg*) is unambiguously to be recognized in both curves.

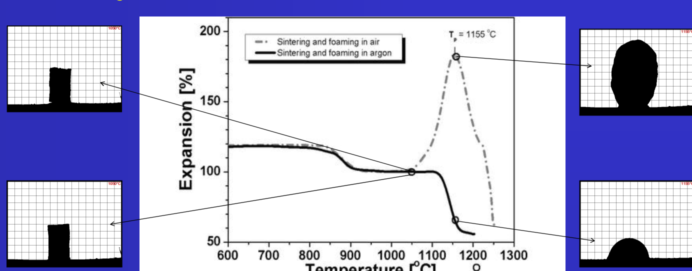

Figure 2: Typical HSM curves of the thermal behaviour of pressed iron-rich glass-ceramic samples in air and argon

Te innovative point of current research is, besides the use of the applied method of sinter-crystallization toward the production of sintered glass-ceramic materials, the use of a controlled environment (air and inert atmosphere) during the synthesis of sintered glass-ceramics.

The effect of the environment on sintering has been unambiguously determined as it is shown in Figure 1a, 1b and 2: the onset of the sintering process in argon is shifted to lower temperatures. It starts earlier, and the degree of structural densification is considerably higher than the one in air. As it is evident from EPR measurements the ratio of Fe2+/Fe3+ is strongly drawn to the right. This is a prerequisite for viscosity decrease due to a predominant presence of Fe3+ (it doesn't contribute to the sintered material's crystal lattice) in the parent glass.

The effect of the rate of heating on the sintering behavior of newly obtained glass-ceramic materials has been and is currently being subject of detailed investigation by the authors. The most important feature is, besides the kinetic shift in temperature, the influence of the linear heating rate on the degree of sintering. Thus different new glass-ceramic materials can be obtained as it is shown in Figure 3. Variations of the heating rate from 2 to 30 degrees per minute lead to approx. 10 % variations in the degree of sintering.

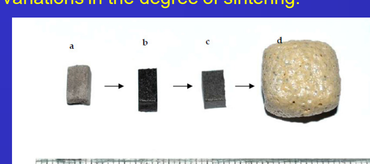

Figure 3: Photos of: a. Green sample; b. Sintered glass-ceramics; c. Auto-glazed material; d. Glass-ceramic foam

Conclusions

In presented investigation the authors have shown the possibilities for carrying out successful synthesis and kinetic investigations of the process of sintering, depending on the firing programs and the applied atmosphere of well-sintered glass-ceramics and eventually glass-ceramic foams. It was shown that synthesis in a mixed air/argon environment leads to the production of materials with higher degree of sintering.

If properly engineered, the practice and experience show that a glassy-crystalline material with high density can be obtained, which can be considered as more than a satisfactory result.

One can thus further summarize that utilizing the HSM technique for the sake of synthesis of well-sintered glass-ceramic materials and foams and for the exact tuning of an appropriate thermal treatment is the right approach.

The authors express thanks for the financial support under the Contract KP-06-H27/14 – 2018 (BSF) and under the Project BG05M2OP001-1.002-0019 "Clean technologies for sustainable environment – waters, waste, energy for circular economy".